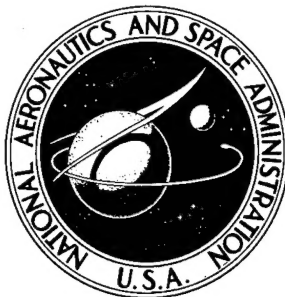
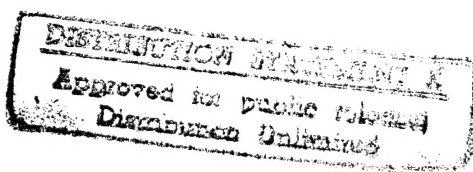


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EFFECTS OF SPACE ENVIRONMENT FACTORS
ON THE MECHANICAL, PHYSICAL, AND
OPTICAL PROPERTIES OF
SELECTED TRANSPARENT ELASTOMERS

by Jerry G. Williams and Joseph H. Judd

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Hampton, Va. 23365

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THE MECHANICAL, PHYSICAL, AND OPTICAL PROPERTIES
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SUMMARY

An experimental investigation was conducted on four different transparent elastomers (silicone, urethane, ethylene-propylene, and isoprene rubbers) which are candidate matrix materials for flexible windows on spacecraft. The material mechanical properties were measured over a temperature range of from -100°C to $+100^{\circ}\text{C}$ and after exposure to vacuum, vacuum-heat, and air-heat environments for a period of 30 days. Light transmission through these materials for wavelengths from 230 to 2500 nm was also determined after exposure to environmental conditions. In addition to mechanical and optical properties, measurements were made on specimens to determine vacuum weight loss, gas permeability, the degrading effects of ultraviolet-vacuum exposure, and thermal degradation characteristics.

The test results indicated that none of the materials would individually maintain in space the mechanical, physical, and optical properties necessary for a satisfactory window matrix material. The silicone rubbers possessed satisfactory mechanical strength over the temperature range and excellent resistance to environmental conditions, but had tear strength and tear propagation resistance too low to be used for manned spacecraft. Urethane rubber showed the best mechanical strength and tear resistance, but had a narrower useful temperature range and marginally acceptable optical properties. The mechanical and optical properties of ethylene-propylene rubber and isoprene rubber were severely reduced after exposure to the air environment at elevated temperatures. Thermal analysis measurements showed that all the elastomers were flammable in 100-percent oxygen at a pressure of 100 kN/m^2 .

INTRODUCTION

In order to improve the safety and utility of manned space structures and to provide a method for direct visual reference and observation, viewing ports or windows are required. The concept of a flexible window or observation dome has been investigated

at the Langley Research Center as part of an expandable space structures program. (See refs. 1 and 2.) Because of potential advantages in packaging and resistance to impact damage, the flexible observation port may also find application for rigid manned space structures. The concept described in references 1 and 2 utilizes a composite construction consisting of a rectangular grid of high-strength filament groups encapsulated in a flexible, transparent matrix. Since the matrix is simultaneously exposed to both the space environment and the cabin environment, it is necessary to determine the effect of these environments on the mechanical, physical, and optical properties of candidate matrix materials. Required properties of a foldable window matrix are: optical clarity, adequate mechanical strength at reasonably low strain levels, good abrasion and tear resistance, flexibility, good bonding to fiber reinforcements, fire resistance, low evolution of gases, and low permeability.

The contents of this report describe the results of an experimental investigation on four different flexible transparent elastomers (silicone, urethane, ethylene-propylene, and isoprene rubbers) to determine their suitability as window matrix material for spacecraft. Mechanical, physical, and optical properties were measured at ambient laboratory conditions (22° C and 100 kN/m² pressure) after exposure to test conditions which simulated elements of the space environment (air heat, vacuum, and vacuum heat). Mechanical properties were also measured "in situ" at elevated and low temperatures and thermal degradation characteristics were determined at elevated temperatures in 100-percent oxygen and 100-percent nitrogen atmospheres.

MATERIALS DESCRIPTION

Composition

For the present study, five flexible transparent elastomers were selected from four different types of polymers. These included silicone (from two manufacturing sources), urethane, ethylene-propylene, and isoprene rubbers. These polymers are generally considered elastomers, although ethylene-propylene and urethane must also be considered as viscoelastic materials.

Silicone.- Silicone 1 and silicone 2 are dimethyl silicone rubbers (poly(dimethylsiloxane)) of the type called room temperature vulcanizing (RTV). The RTV silicone rubbers are characterized by optical clarity, retention of mechanical properties over a broad temperature range, and excellent resistance to environmental degradation.

Urethane.- The transparent polyurethane rubber used in the present study was a polyether urethane which is formed by the reaction of bifunctional polyols (poly-(tetramethylene adipate) and 1,4-butanediol) and diphenylmethane diisocyanate. Outstanding

properties of the urethane include abrasion resistance, high tear strength, retention of mechanical properties at low and elevated temperatures, and hydrolytic stability.

Ethylene-propylene.- The ethylene-propylene terpolymer of this study was a hydrocarbon rubber. The linear saturated polymer is formed by the polymerization of ethylene and propylene with a diene monomer such as dicyclopentadiene. The material is characterized by good mechanical strength, tear resistance, and optical clarity.

Isoprene.- Isoprene is a synthetic elastomer with the same composition as natural rubber, cis-1,4-polyisoprene. The material has high elongation capability.

Specimen Preparation

Formulations.- The basic polymers used in the present study were off-the-shelf formulations selected from experience and vendor literature based on apparent mechanical, physical, and optical properties. Wherever necessary, appropriate additives were incorporated into the polymers to improve clarity, curing characteristics, or physical properties. The exact formulations of the test materials are given in table I. Small variations in the basic material composition, polymer formulation, curing temperature,

TABLE I.- ELASTOMER FORMULATION

| Material | Composition, parts by weight | | | | |
|--|---------------------------------|---------------------------------|--|--|--|
| | Silicone 1 | Silicone 2 | Urethane | Ethylene propylene | Isoprene |
| Dimethyl silicone rubber, RTV 615,* Part A | 100 | | | | |
| Dimethyl silicone rubber, RTV 615,* Part B | 10 | | | | |
| Dimethyl silicone rubber, Sylgard* 184, Part A | | 100 | | | |
| Dimethyl silicone rubber, Sylgard* 184, Part B | | 10 | | | |
| Polyester urethane, Estane* 5740 x 140 | | | 100 | | |
| Ethylene propylene terpolymer, Nordel* 1040 | | | | 100 | |
| Polyisoprene 309* | | | | | 100 |
| Oxirone* 2000 | | | | 2 | |
| Buton* 150 | | | | 3 | |
| Cab-o-sil* | | | | 20 | |
| Varox* (liquid) | | | | | 1.25 |
| Cyasorb* UV-9 | | | | | .6 |
| Antioxidant* 2246 | | | .25 | | |
| Fabrication process | Cast, cured 2 hours at 71° C | Cast, cured 2 hours at 71° C | Molded, cured 15 minutes at 177° C | Molded, cured 30 minutes at 166° C | Molded, cured 20 minutes at 149° C |

*Registered trademark.

and curing time can produce large differences in the mechanical properties of polymeric materials. However, in most cases measurements were made on specimens from the same batch.

Specimens.- The candidate polymers were either molded or cast into square sheets approximately 38 cm in width using 3.2-mm-thick picture frame molds. Departures from the nominal thickness were due to side effects caused by either molding pressure or mold thickness. Specimen sheet thickness ranged between 2.7 mm and 3.9 mm. To obtain a smooth surface, the test sheets were either cast against a highly polished chrome-plated steel plate having a 1/2 root-mean-square finish or molded in a frame containing ferro-type surface plates.

Silicones 1 and 2 had a clear appearance and the ethylene-propylene rubber and isoprene rubber were clear to milky. The urethane rubber had a pronounced yellow color. When the specimen surfaces were examined in reflected light, the silicone rubbers appeared to be smooth with a glossy surface, whereas all other materials had smooth surfaces with a slight matte appearance.

Specimens for uniaxial load tests were cut from the polymer sheets by a steel dumb-bell die "E" described in reference 3. All cut specimens were examined for surface irregularities, and unsatisfactory specimens were discarded.

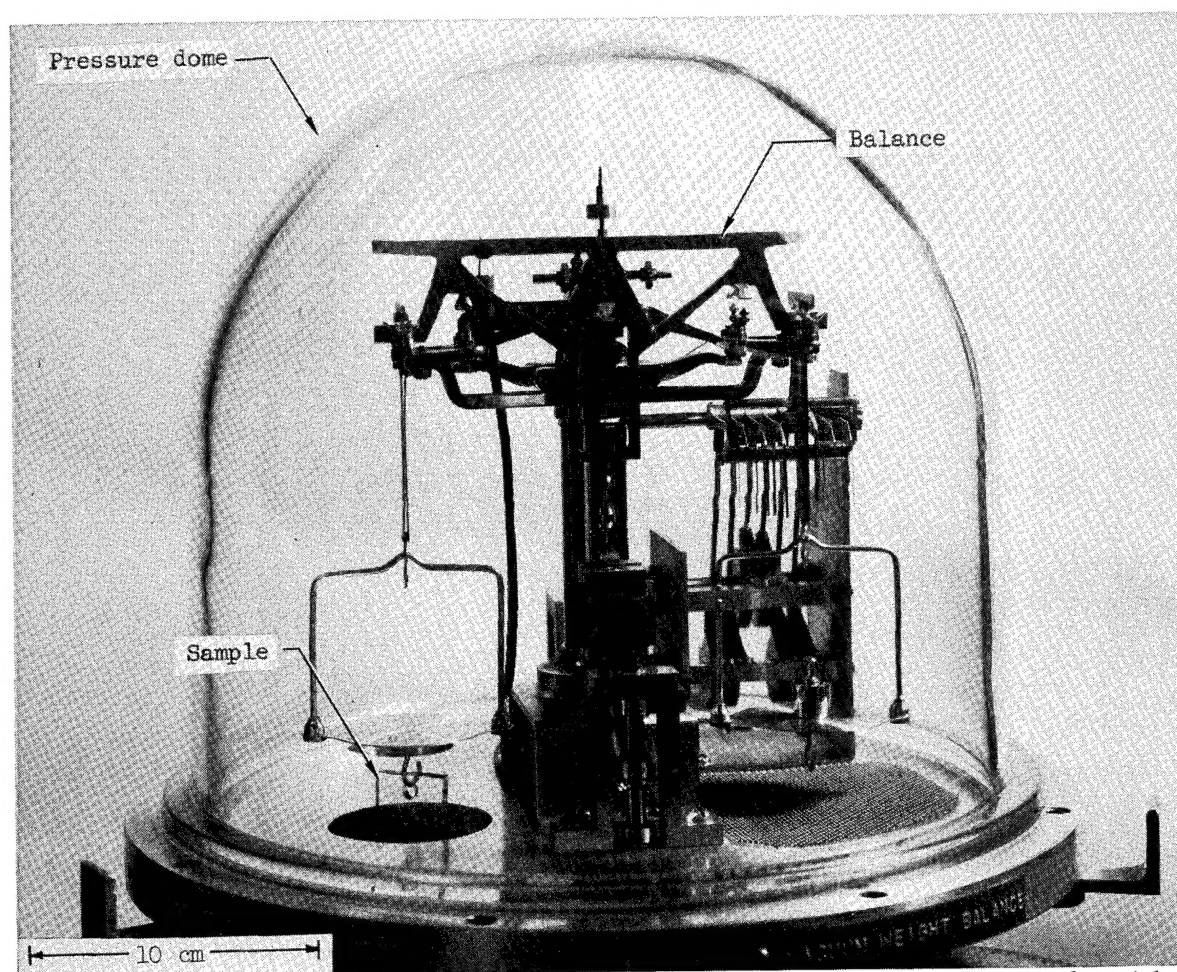
APPARATUS

Environmental Systems

Vacuum system.- Polymer specimens were subjected to low pressures at ambient laboratory temperatures in two vacuum systems: an ultrahigh-vacuum system and a vacuum weight system.

The ultrahigh-vacuum system consisted of a 0.466-meter diameter and 0.761-meter high stainless-steel belljar seated on a stainless-steel plate and a pumping system which is mounted below the plate. The major elements of the pumping system were a mechanical pump, an ion pump, and a titanium sublimation pump. Bakeout heaters and water cooling were utilized as aids to pump the system to a pressure of approximately 67 nN/m^2 in approximately 20 hours. A thermocouple vacuum gage tube was installed between the isolation valve and the mechanical pump, and two ionization gages were located in the tee below the belljar. A stainless-steel rack was positioned on the belljar mounting plate. Test specimens were suspended from the rack by chrome-plated spring clips.

The vacuum weight system is shown in figure 1. The balance, which is described in a following section, was mounted in a glass belljar of 0.01 m^3 volume and the specimen was suspended in a stainless-steel cylinder below the mounting plate. The pumping

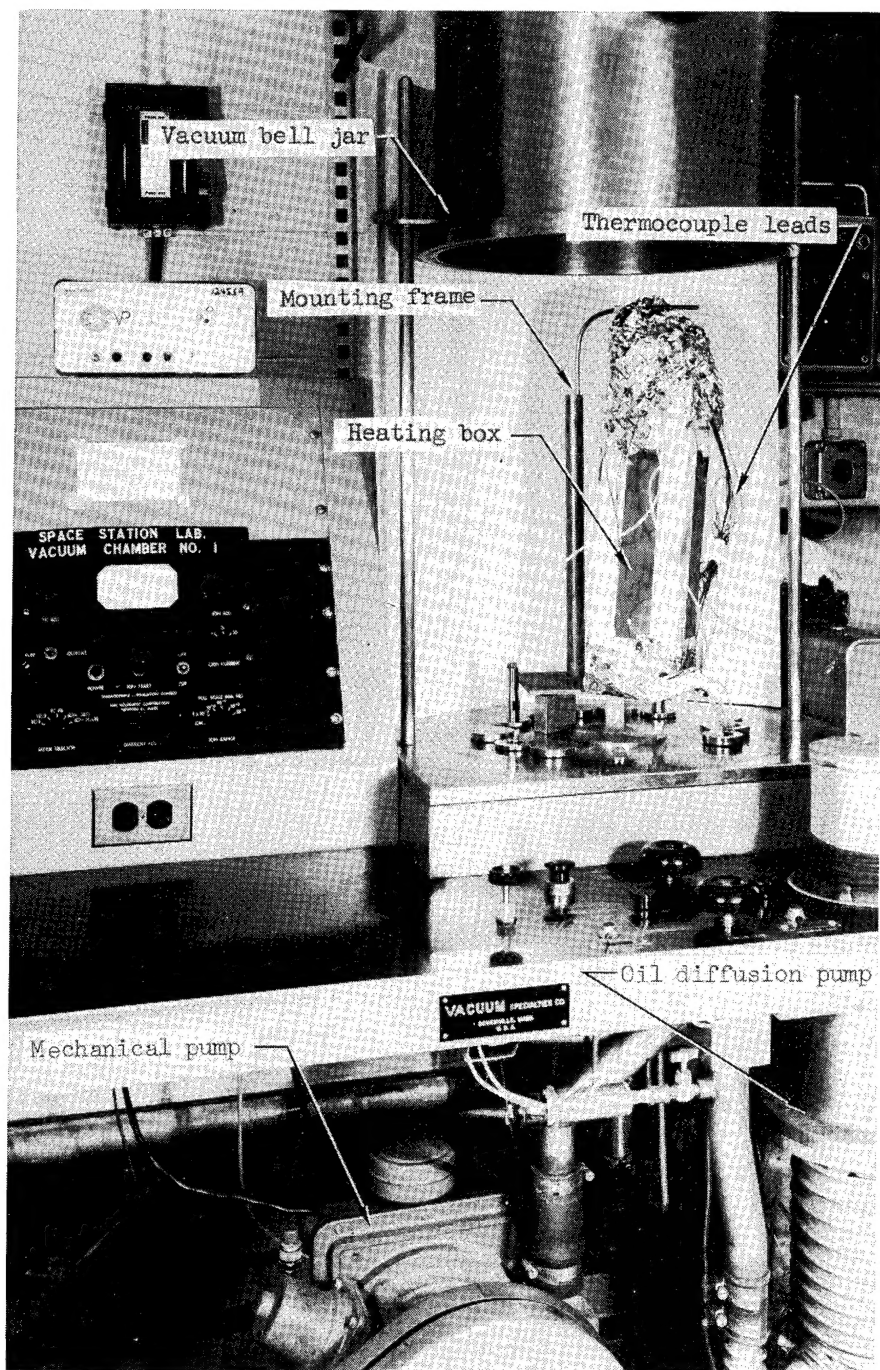


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Figure 1.- Photograph of the vacuum weight system.

system consisted of an oil diffusion pump backed by a mechanical pump. In order to reduce backstreaming of the silicone pump oil, the pump was cooled by a fluid at an inlet temperature of -10°C . Pressure was measured by an inverted ion gage.

Vacuum-heat system.- The system used in the vacuum-heat exposure test is shown in figure 2. The vacuum pumping system consisted of an oil diffusion pump backed by a mechanical pump and was capable of maintaining a "no sample" pressure below $65\ \mu\text{N}/\text{m}^2$. An ionization gage and an emission-regulated ion gage control were used for the measurement of the pressure inside the stainless-steel belljar. The belljar was 0.45 meter in diameter and had an internal height of 0.76 meter. Heating was provided by a set of four rectangular heating elements (38.2 cm by 11.5 cm) joined together to form a box. After the specimens were suspended inside the box, the ends were loosely closed off with aluminum foil and fiber-glass tape. This procedure yielded a thermal black box, and yet enabled outgassing products to be collected by the pumping system. Seven thermocouples spaced throughout the volume of the thermal black box monitored the temperature.

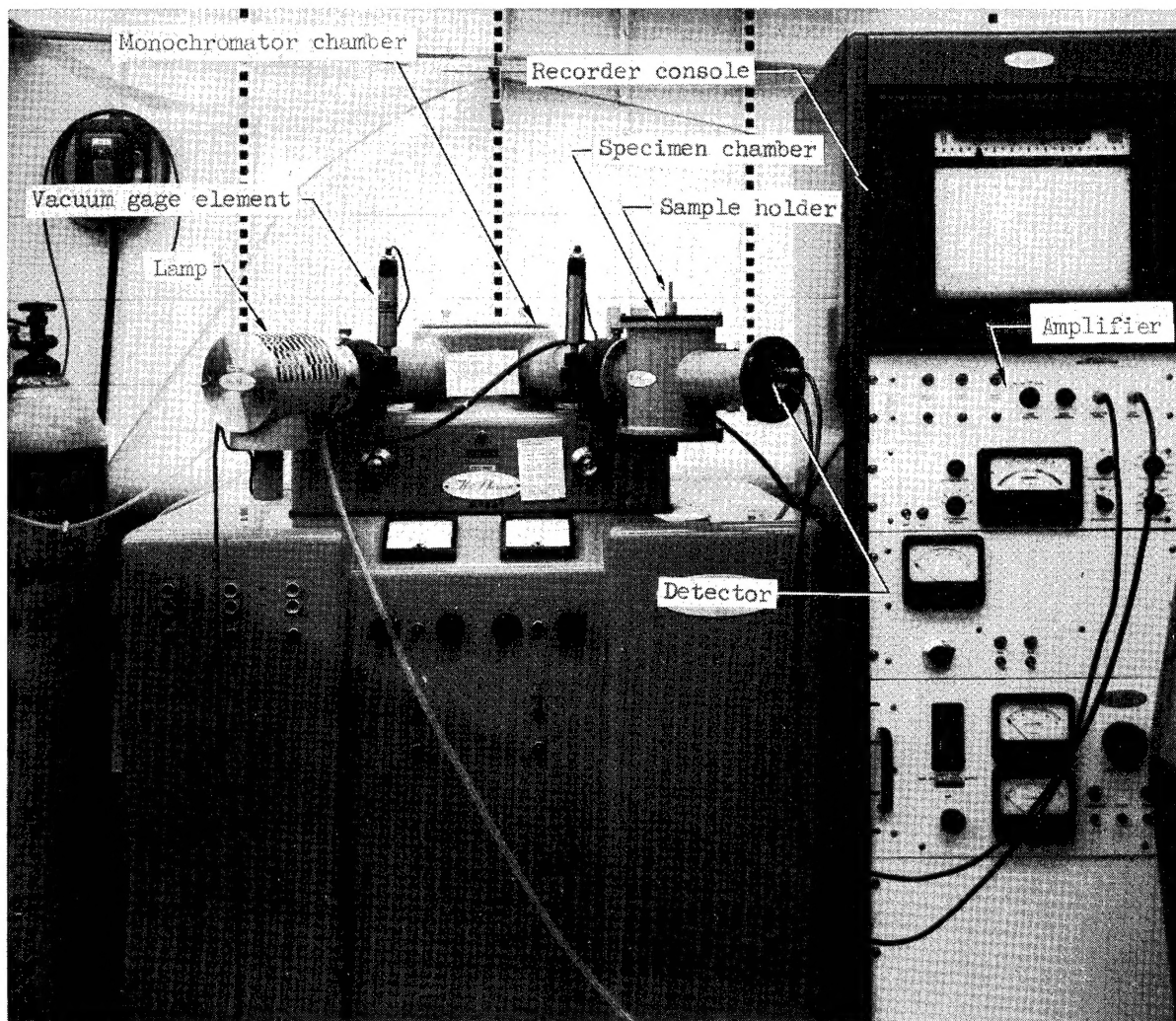


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Figure 2.- Photograph of the vacuum-heat system.

Ultraviolet-vacuum system.- The major elements of the system that was used to expose the transparent polymers to ultraviolet radiation were a light source, a scanning monochromator, a detector, an amplifier, and a recorder. The radiation was emitted by a capillary discharge light source which is capable of handling 1000 watts of energy continuously and up to 8000 watts intermittently. A photograph of the system is presented in figure 3(a).

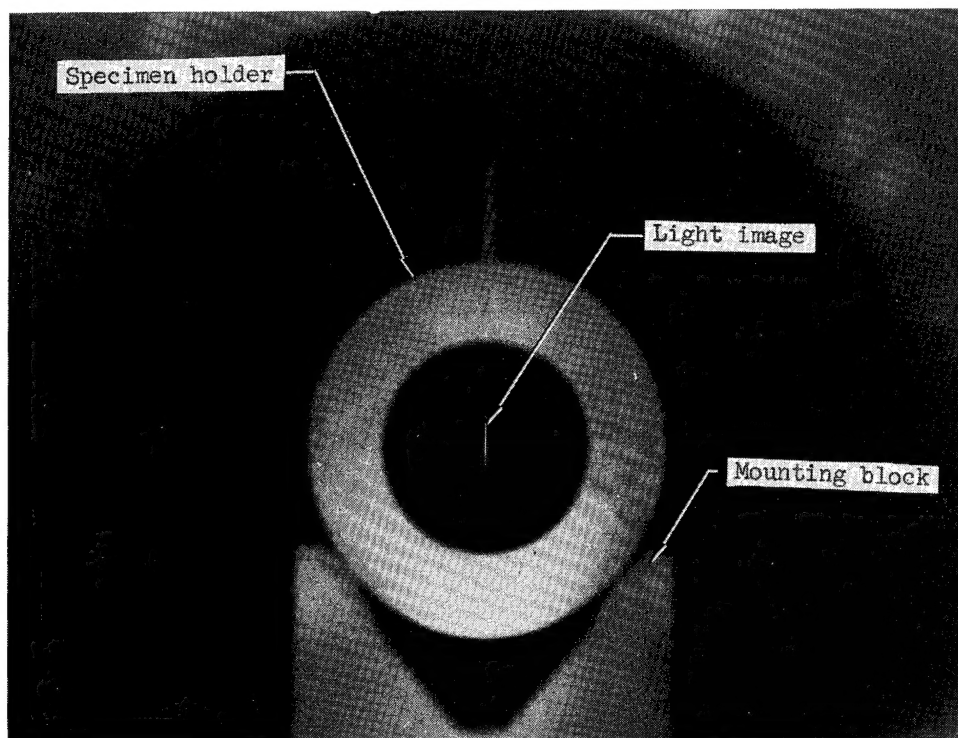
A ring-shaped specimen holder was mounted on a slender rod which could be used to raise and lower the specimen relative to the radiation field. At the exposure position, the holder was located on a V-block as shown in figure 3(b).



(a) Major system elements.

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Figure 3.- Ultraviolet-vacuum system.



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(b) Specimen mount and exposure source in test chamber.

Figure 3.- Concluded.

Oven.- A gravity-convection-type oven was used to heat the specimens for the air-heat exposure test. Six chromel-alumel thermocouples spaced throughout the oven were used to monitor the temperature.

Universal test machine environmental chamber.- A portable environmental chamber was used with the uniaxial load machine to measure the tensile properties of specimens under a controlled temperature condition. The chamber included an enclosure around the test specimen, a gas circulation system, a heating and cooling system, and a temperature control instrument. The environmental chamber used liquid nitrogen for cooling and the gaseous nitrogen evolved from the coolant was discharged directly into the chamber. The grip displacement allowed by the constraints of the chamber was 7.7 cm. Accuracy of the temperature control instrument was approximately $\pm 3^{\circ}\text{C}$. The actual test temperature recorded was measured by a thermocouple located close to the middle of the specimen (within approximately 1 cm). The thermal gradient within the test chamber was within $\pm 4^{\circ}\text{C}$.

Instrumentation and Measurement Systems

Vacuum balance.- A balance, sensitive to $9.81\ \mu\text{N}$, was installed in the vacuum weight system. A strip chart recorder which worked on a null basis through a potentiometer recorded the specimen weight. The system had a sensitivity of 98 mN.

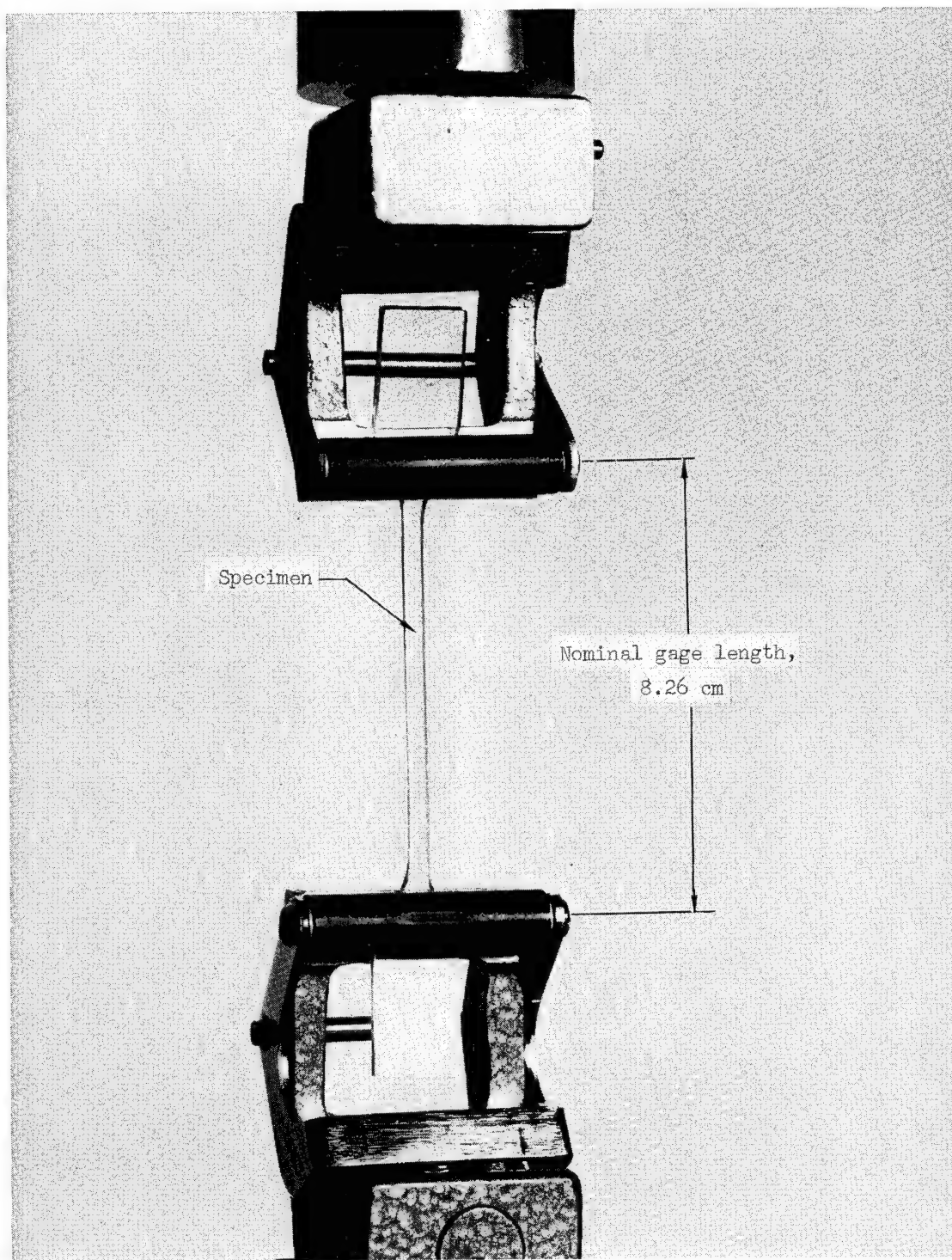


Figure 4.- Test fixture clamps.

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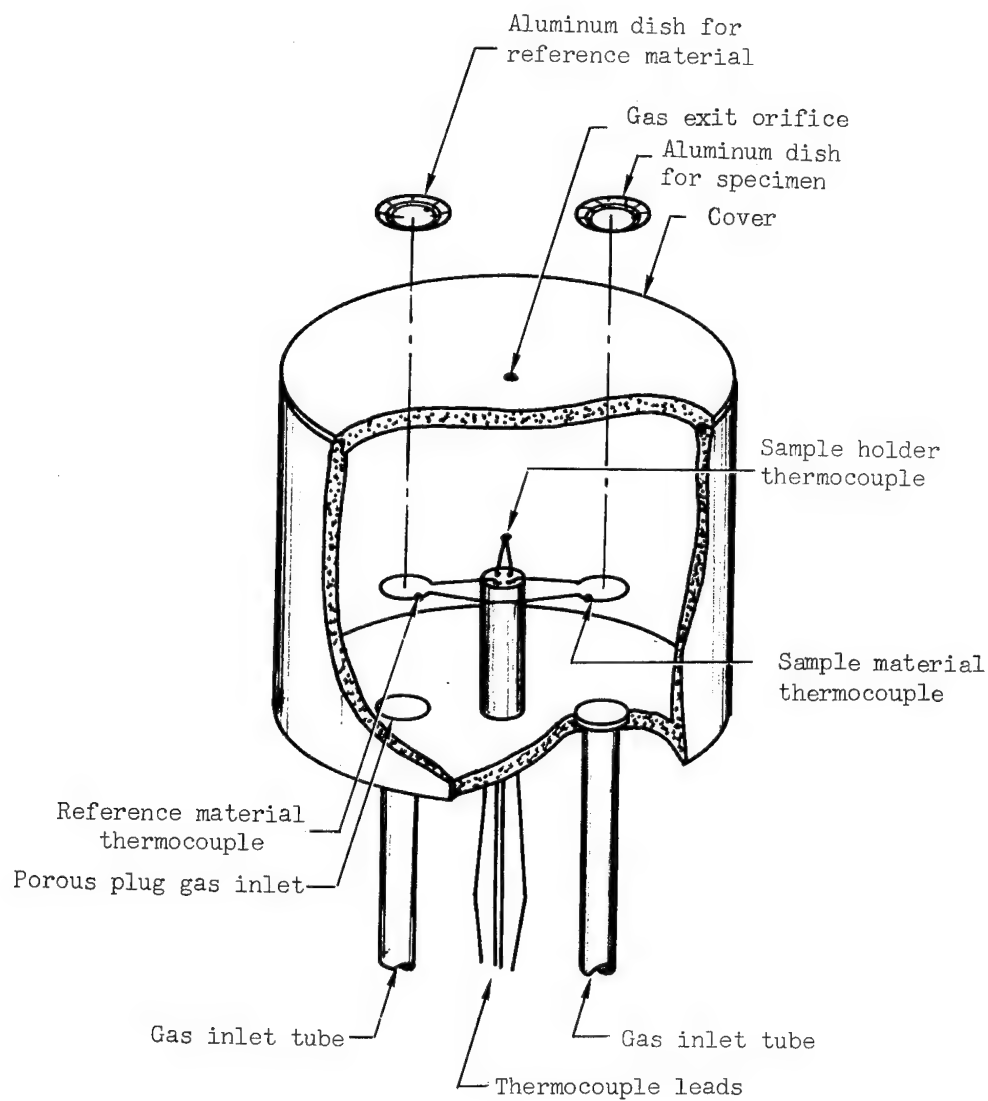
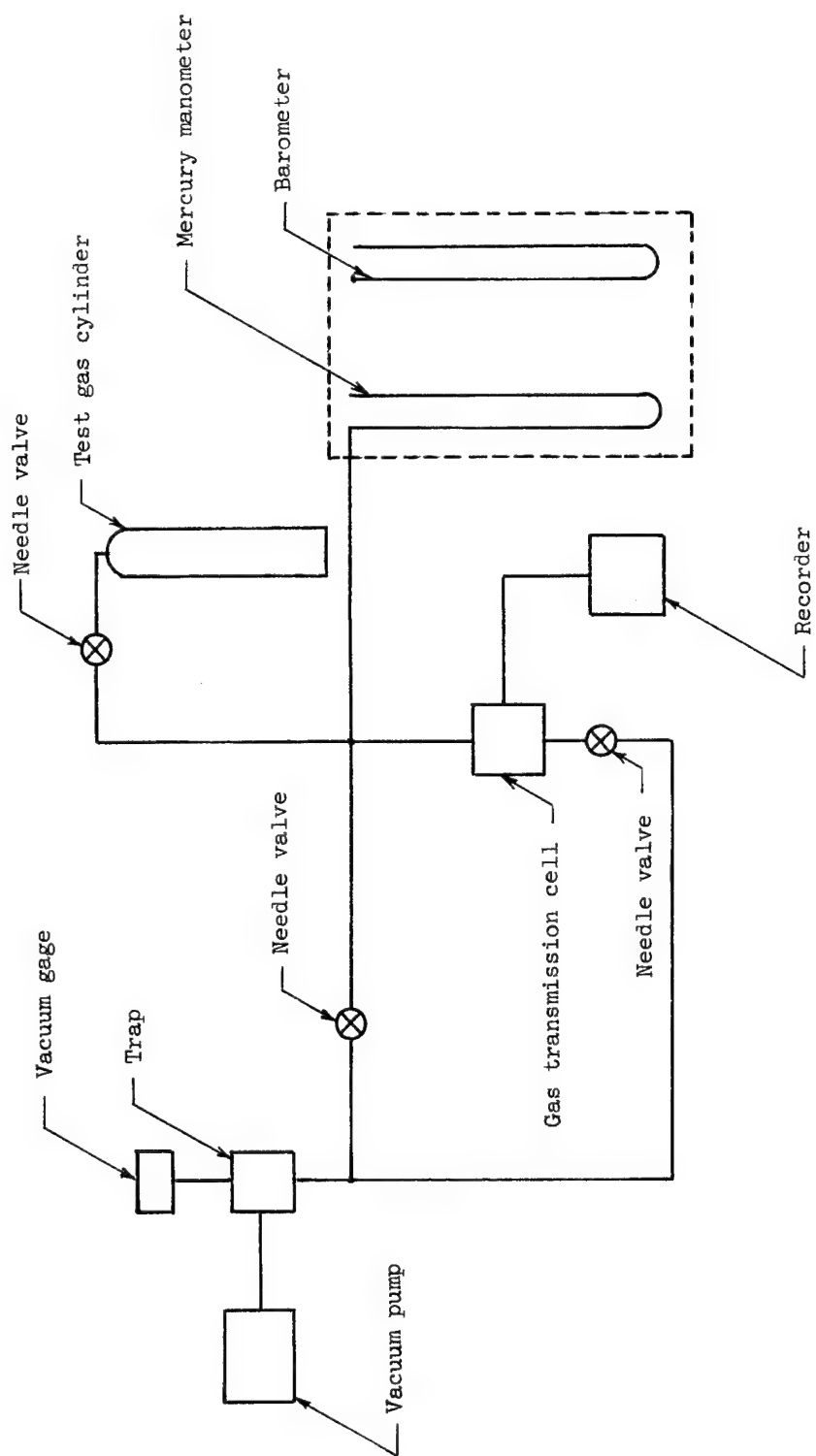
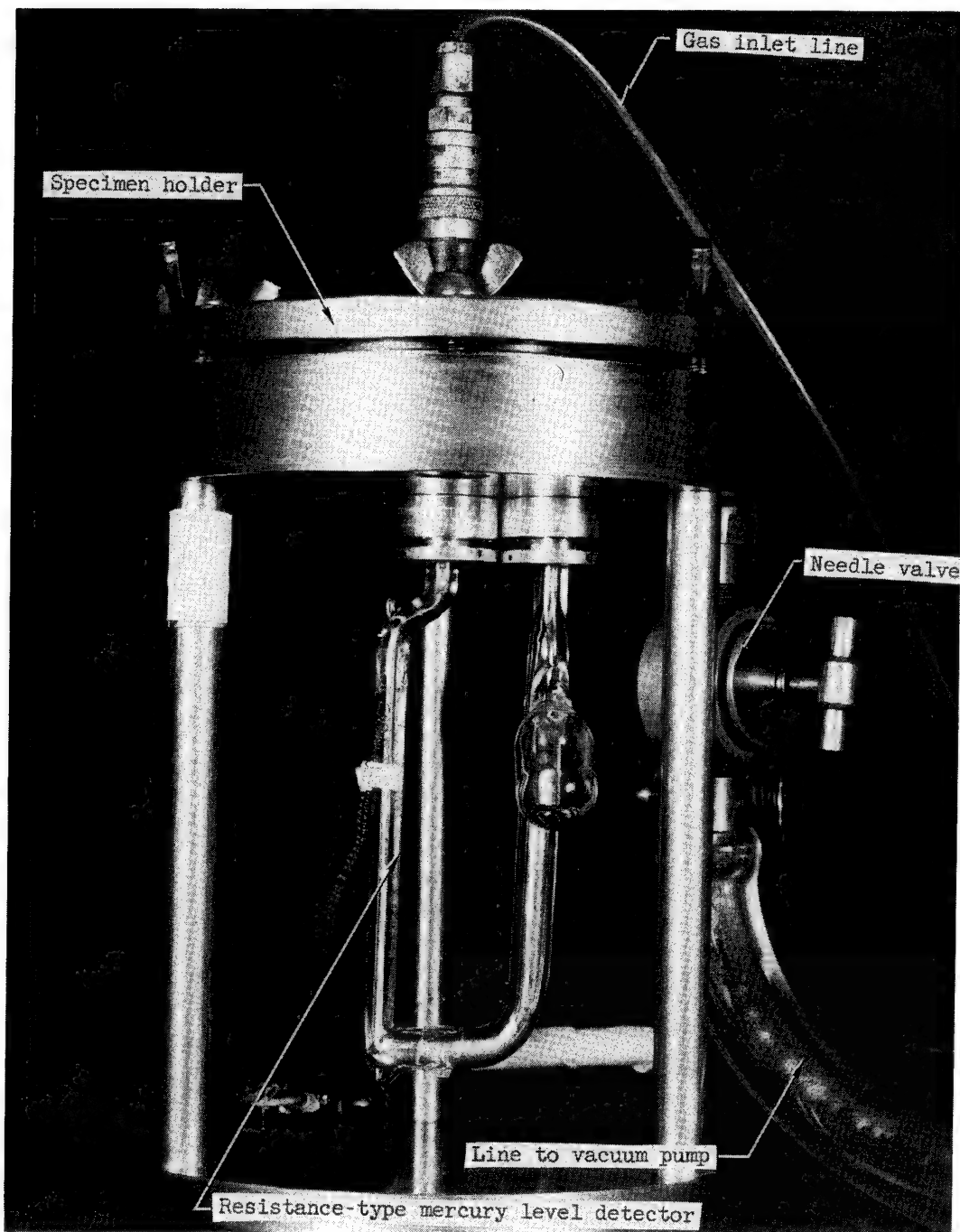


Figure 5.- Sketch of the differential thermal analysis (DTA) sample holder.



(a) Schematic of gas transmission measurement system.

Figure 6.- Gas transmission measurement apparatus.



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(b) Photograph of the Dow gas transmission cell.

Figure 6.- Concluded.

Uniaxial load instrumentation.- The mechanical properties of the elastomers were obtained by the use of a standard uniaxial load machine. (See ref. 4.) An electronic integrator was used to determine the area under the curve for the variation of load against deformation. The accuracy of the integrator is within ± 1 percent. The accuracy of the load-detection system is independent of the range in use and is better than ± 0.5 percent. A photograph of a representative specimen in the test fixture clamp is presented in figure 4.

Thermal analysis system.- The differential thermal analysis system (DTA) which was used for the measurements presented in this paper consisted of a recorder-controller, a furnace platform, a furnace, and a sample holder. A sketch of the sample holder is presented in figure 5. The reference temperature in the sample holder was measured by a chromel-alumel thermocouple, and the temperature difference between the sample and a reference material by ring-type platinum thermocouples. Calcined alumina was used as the neutral body reference material. The programmed heating rate used in these tests was 10°C per minute.

Gas transmission measurement system.- The diffusion of gases through sheets of the flexible transparent polymers was obtained by using a system composed of a Dow gas transmission cell, manometers, vacuum gage, vacuum pump and trap, regulating valve, and barometer. A schematic of the component arrangement is presented in figure 6(a) and a photograph of the cell is presented in figure 6(b). A description of the equipment is presented in reference 5. The test specimens were circular and had areas of 65.2 cm^2 .

TESTS AND MEASUREMENTS

Environmental Exposure of Specimens

The procedure used in the environmental exposure of the specimens involved specimen inspection, cleaning, and weight measurement, followed by insertion of the specimens into the respective environmental system for approximately 30 days. Specimens were exposed to air at a temperature of 100°C , the low pressure of a vacuum system at ambient laboratory temperatures of approximately 22°C , and the low pressure of a vacuum system at a temperature of 100°C . Pressure measurements in the vacuum chambers were read and recorded by the test operator several times daily. The temperature adjacent to the specimens and the ambient laboratory temperature were automatically recorded. Immediately after completion of the exposure sequence, the specimens were withdrawn from the chamber. Ambient laboratory temperature and relative humidity were recorded, and specimens were again weighed.

Vacuum Weight Tests

The weight of the flexible transparent polymers was continuously measured in a vacuum for 30 days. After initial specimen weight determination on an analytical balance, clean specimens were suspended by a hook from one arm of the balance and counter-weights were positioned on the balance pan to approximate closely the measured weight. The vacuum system was then pumped down and the balance reading was noted. This value was taken to be the initial specimen weight. After 30 days the specimen was removed from the system and immediately weighed on the analytical balance and the value compared with the value obtained on the vacuum weight balance. Agreement to within ± 1 milligram was usually obtained. Test specimens were approximately 15 cm long, 2.5 cm wide, and 0.28 cm thick.

Weight changes were also determined for tensile specimens which were subjected to environmental exposure (air-heat, vacuum, and vacuum-heat). Measurements were made with an analytical balance in air before and after exposure.

Ultraviolet-Vacuum Exposure

The test specimen geometry used in the ultraviolet-vacuum exposure tests was cut by a die E described in reference 3. After specimen cleansing by water and ethyl alcohol, the specimen was positioned horizontally in the ring mount and centered at the ring axis so that the radiation beam bisected the specimen. The ring mount was raised manually out of the path of the radiation beam, the vacuum system was pumped down, and the lamp was started. The wavelength was set on the monochromator manually by observation of the detector output and the specimen was lowered into the test position. The detector output was continually recorded and a periodic check was made for peak drift. Specimen exposure conditions are shown in table II.

TABLE II.- ULTRAVIOLET-VACUUM EXPOSURE CONDITIONS

(a) Exposure wavelength of 134.3 nm using H₂ in lamp

| Elastomer | Main chamber pressure, mN | Exposure, hr |
|------------|---------------------------|--------------|
| Silicone 1 | 16 to 8.7 | 6 |
| Urethane | 27 to 5.3 | 6 |
| Isoprene | 5.9 to 1.7 | 6, 24 |

(b) Exposure wavelength of 1216 Å using He in lamp

| Elastomer | Main chamber pressure, mN | Exposure, hr |
|------------|---------------------------|--------------|
| Silicone 1 | 13 to 8.0 | 48 |
| Silicone 2 | 4.0 to 2.8 | 24 |
| Urethane | 4.3 to 1.4 | 24, 48 |

After completion of the exposure and removal of the specimen from the system, a microscopic survey was conducted on the exposed specimen area with the test specimen relaxed and with approximately 100-percent elongation.

Mechanical Properties

Uniaxial loading.- Tensile load tests were conducted in the ambient laboratory environment and in a closed thermal environmental chamber. Stress and strain measurements were made according to the procedure of reference 3. In data reduction, the decreases in cross-sectional area due to lateral strain and the increases in cross-sectional area due to thermal expansion of the material were neglected. The initial grip separation distance was maintained at 8.25 cm and, except where noted on the presented data, had a jaw displacement rate of 25.40 cm/min. Particular care was used in insertion of the elastomer specimens into the grips to avoid lateral bending of the specimens between the grips and to minimize preloading deformations. Where possible, tests were conducted to failure. Isoprene rubber, however, had such a high maximum strain capability that the displacement capacity of the uniaxial loading machine was exceeded. The grip motion of the loading machine was further restricted by the internal dimensions of the thermal environmental chamber, and most of the elastomers could not be tested to failure in elevated and low temperature tests.

Gage-length calibration for tensile tests.- The test procedure of reference 3 requires that elongation measurements be made between bench marks located on the constant cross-sectional area of the die-cut sheet elastomers. In the present series of uniaxial loading tests, this experimental technique proved to be difficult because the elastomers did not accept marking readily and accurate measurements could not be made when the specimens were tested within the thermal environmental chamber. In addition, it was believed that exposure to the various environments would probably reduce the legibility of the bench marks and introduce the potential of specimen damage at the marks. For these reasons it was decided to use the distance between the grips as the gage length to calculate a nominal strain for comparative purposes.

A test was conducted to establish the difference in strain as determined by these two types of gage-length measurements. In this test two thin line elements of white silicone rubber were applied approximately 5.08 cm apart on the constant cross-sectional area of the die-cut specimens. A motion-picture camera was used to photograph the mark displacement, a reference scale, and a stopwatch. The time variation of bench-mark displacement was measured by projecting the film by a time and motion study projector. The time measurement was used to correlate the measured elongation with the crosshead displacement of the load machine. A graphical representation of the results for the test materials is shown in figure 7. Measured strain is based on line elements

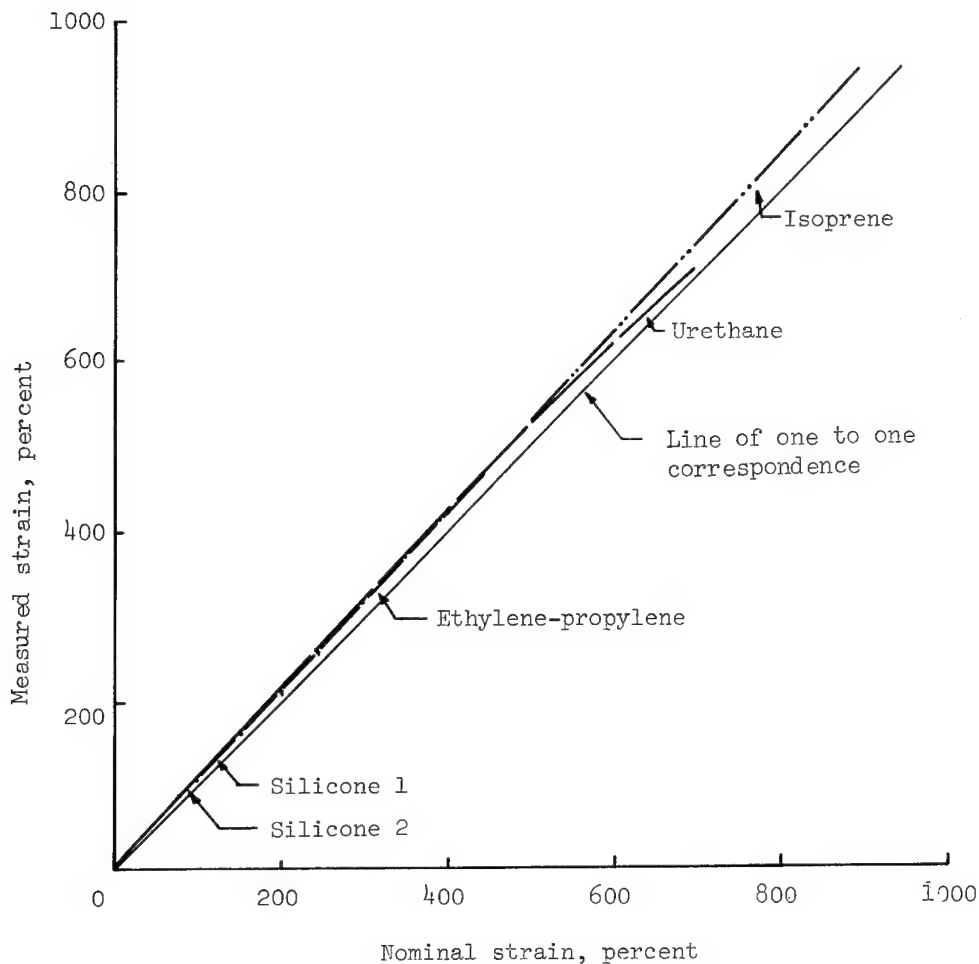


Figure 7.- Gage-length calibration for the elastomers.

marked on the specimen and nominal strain on the crosshead separation. It is seen that the maximum variation is on the order of 5 percent.

Stress relaxation.- Measurements of the elastomer stress relaxation response were made on the uniaxial loading machine on each specimen for a period of 1000 seconds. Ambient and elevated temperature tests were conducted in air, and reduced temperature tests were in a nitrogen-rich atmosphere. Grip separation distance was used as the gage length and the initial separation was 8.25 cm. The test specimens were extended to an initial strain (see table III) at a grip displacement rate of 25.4 cm/min. The stress at any time divided by the corresponding strain is defined as the relaxation modulus.

Tear tests.- Measurements of the tear resistance of the elastomers were made by use of the procedure described in reference 6. Specimens were cut from the elastomer sheets by a D1004 die. Specimens were installed in grips on the uniaxial loading machine and loaded to failure. Ten specimens of each material were tested. Specimen thicknesses

TABLE III.- INITIAL STRAIN FOR STRESS RELAXATION TESTS

| Elastomer | Nominal strain, percent, at indicated temperature of - | | | | | | | |
|--------------------|--|-------|-------|-------|-------|-------|-------|--------|
| | 100° C | 66° C | 40° C | 22° C | 22° C | 22° C | 0° C | -20° C |
| Silicone 1 | 84.4 | 82.6 | 119.4 | 83.2 | 98.5 | 129.0 | 129.6 | --- |
| Silicone 2 | 57.3 | 46.2 | (*) | 46.2 | 84.7 | (*) | ---- | --- |
| Urethane | 47.3 | 254.3 | 118.5 | 169.0 | 369.0 | 493.0 | 46.2 | 9.2 |
| Ethylene-propylene | 168.5 | 137.9 | 203.0 | 354.0 | 449.0 | 173.0 | 173.0 | 8.6 |
| Isoprene | ---- | 190.5 | 276.0 | 123.0 | 431.0 | (*) | ---- | --- |

*Specimens failed during elongation or during relaxation.

were within the ± 10 percent specified by the ASTM procedure, both for individual elastomers and for the elastomers as a group.

Physical Properties

Permeability.- Measurements of the permeability of the elastomers to oxygen and nitrogen gases were made by following the procedures of reference 7. For materials with very fast gas transmission rates, the time required to increase the pressure in the manometer by 5.0 cm of mercury was recorded manually. Otherwise, standard autographic recording techniques were used.

Differential thermal analysis.- In the differential thermal analysis tests, the potential difference between a thermocouple located beneath an aluminum dish containing the specimen and a thermocouple located beneath an aluminum dish containing the inert reference material was measured as the temperature of the sample holder was increased at a uniform rate from ambient temperature to 500° C. All tests were made by the dynamic atmosphere technique which uses the flow of a gas through the sample holder to entrain and remove effluents which are emitted by the specimen. Each specimen was tested at a pressure of 100 kN/m² in atmospheres of 100-percent oxygen and 100-percent nitrogen.

Optical Measurements

Transmittance.- Test specimens used in transmittance measurements were cleaned prior to environmental exposure in order to remove surface film and dust. In the interval between removal from the environmental chambers and the spectrophotometer tests, each specimen was stored in a sealed polyethylene bag. However, because of the tendency of the materials to collect a static charge during handling, a small amount of dust accumulated on the surfaces.

A spectrophotometer was used for transmittance measurements for wavelengths between 230 nm and 2500 nm. Data presented have been corrected for optical differences between the reference and sample paths.

Optical clarity.- For comparative purposes, photographs were made of a resolution test chart in which a panel of the elastomer was located between the camera and the test chart. (The camera was located 0.305 meter from the specimen and 1.2 meters from the test chart.) Surface reflections were eliminated by darkening the room and directly illuminating the test chart. Specimens were cleaned prior to each test to remove dust.

RESULTS AND DISCUSSION

General Considerations

The exposure of manned spacecraft windows is to the space environment on one side and to the cabin atmosphere on the other side. The test technique for the present investigation was to expose specimens to elements of the space environment and to measure subsequently the characteristic properties in the laboratory at ambient conditions (22° C and 100 kN/m² pressure). Results obtained in this manner may differ from measurements made "in situ." (See ref. 8.) However, for the proposed application, the measured data are considered to be representative of actual conditions.

Properties at Ambient Laboratory Conditions

Mechanical.- Ultimate stress and nominal strain, energy at break, and tear strength are summarized in table IV for each of the five materials. The tear test failure

TABLE IV.- MECHANICAL PROPERTIES OF THE ELASTOMERS IN
AIR AT AMBIENT LABORATORY CONDITIONS*

[Number following \pm is one standard deviation]

| Elastomer | Ultimate strength, MN/m ² | Ultimate nominal strain, percent | Energy at break, joules | Tear strength, kN/m |
|--------------------|--------------------------------------|----------------------------------|-------------------------|----------------------|
| Silicone 1 | 3.15 \pm 0.16 | 165 \pm 13 | 1.323 \pm 0.221 | 5.820 \pm 0.707 |
| Silicone 2 | 3.29 \pm 0.54 | 98 \pm 10 | 0.943 \pm 0.348 | 4.840 \pm 1.110 |
| Urethane | 23.2 \pm 1.8 | 734 \pm 39 | 537.7 \pm 40.1 | 78.70 \pm 1.56 |
| Ethylene-propylene | 12.7 \pm 1.0 | 534 \pm 29 | 260.0 \pm 29.1 | 26.40 \pm 2.43 |
| Isoprene | ** 1.19 \pm 0.05 | ----- | ** 4.84 \pm 0.30 | ** 1.910 \pm 0.210 |

*Average of 10 specimens.

**Measured at strain of 934 percent without failure.

mechanism of the urethane rubber and the ethylene-propylene rubber was characteristic of uniaxial loading since the high-strain capacity of these materials permits material deformation rather than notch or tear propagation.

Representative variations of stress with nominal strain for the elastomers are compared in figure 8. Aside from isoprene rubber which did not break at the maximum test

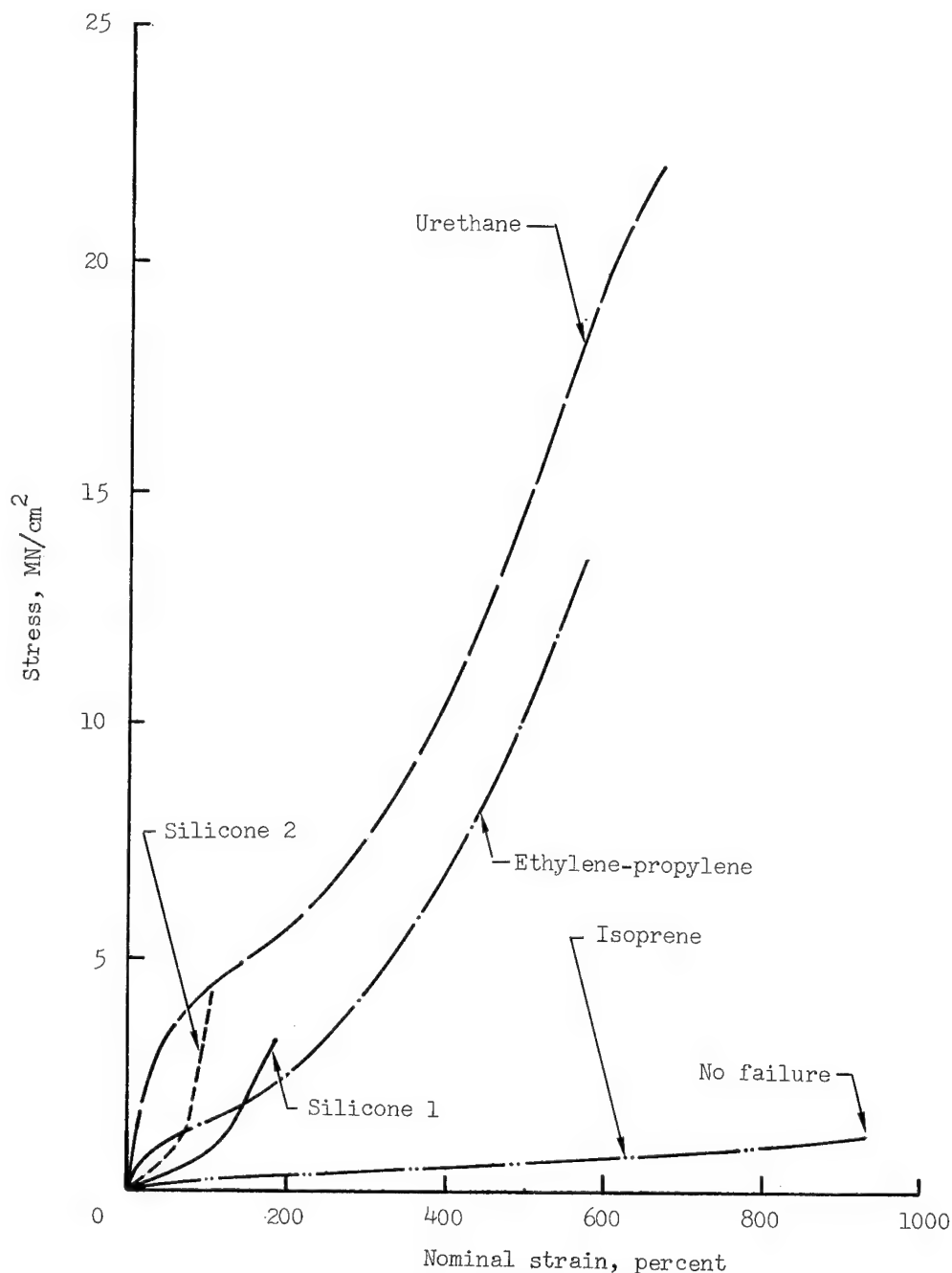


Figure 8.- Representative variation of stress with nominal strain for the elastomers at ambient laboratory conditions.

machine extension, all curves terminate at specimen failure. It is apparent that urethane is mechanically superior to the other elastomers with respect to ultimate strength and modulus.

Permeability.- Measured gas transmission rates of the polymers for 100 percent oxygen and 100 percent nitrogen gases are listed in table V. Permeability of all the materials to oxygen is approximately twice that for nitrogen with the exception of the ethylene-propylene rubber which is approximately three times as great. The lowest permeability values were for the urethane rubber and were an order of magnitude less than the permeability values for either the ethylene-propylene or isoprene rubbers and two orders of magnitude less than those for the silicone rubber materials. For comparison, polyvinylidene chloride, a material with one of the lowest known values for gas permeability, has values of 32×10^{-6} and 6.5×10^{-6} cm³-mm/N-day for permeability to oxygen and nitrogen, respectively. (See ref. 9.) This value is approximately two orders of magnitude less than the permeability for the urethane.

TABLE V.- GAS PERMEABILITY*

[Number following \pm is one standard deviation]

| Elastomer | Average thickness, mm | Permeability, $\frac{\text{cm}^3\text{-mm}}{\text{N-day}}$ | |
|--------------------|-----------------------|--|-----------------------|
| | | Oxygen | Nitrogen |
| Silicone 1 | 3.064 | 0.297 \pm 0.009 | 0.166 \pm 0.014 |
| Silicone 2 | 2.976 | 0.253 \pm 0.014 | 0.126 \pm 0.005 |
| Urethane | 2.979 | 0.00196 \pm 0.00049 | 0.00076 \pm 0.00021 |
| Ethylene-propylene | 3.409 | 0.0142 \pm 0.0013 | 0.0042 \pm 0.0009 |
| Isoprene | 3.189 | 0.0102 \pm 0.0015 | 0.0049 \pm 0.0014 |

*Average for three tests per specimen on three specimens.

Transmittance.- Results of transmittance measurements of radiation in the ultra-violet, visible, and infrared spectrum for the five elastomers are presented in figure 9. These data show that the silicone elastomers possess significantly higher transmittance than the other materials and have a relatively constant value in the visual spectrum (400 nm to 750 nm).

Optical clarity.- Photographs of a test chart taken with no specimen and with each of the five materials located between the camera and the test chart are presented in figure 10. The photographs indicate some local reduction in clarity caused by specimen

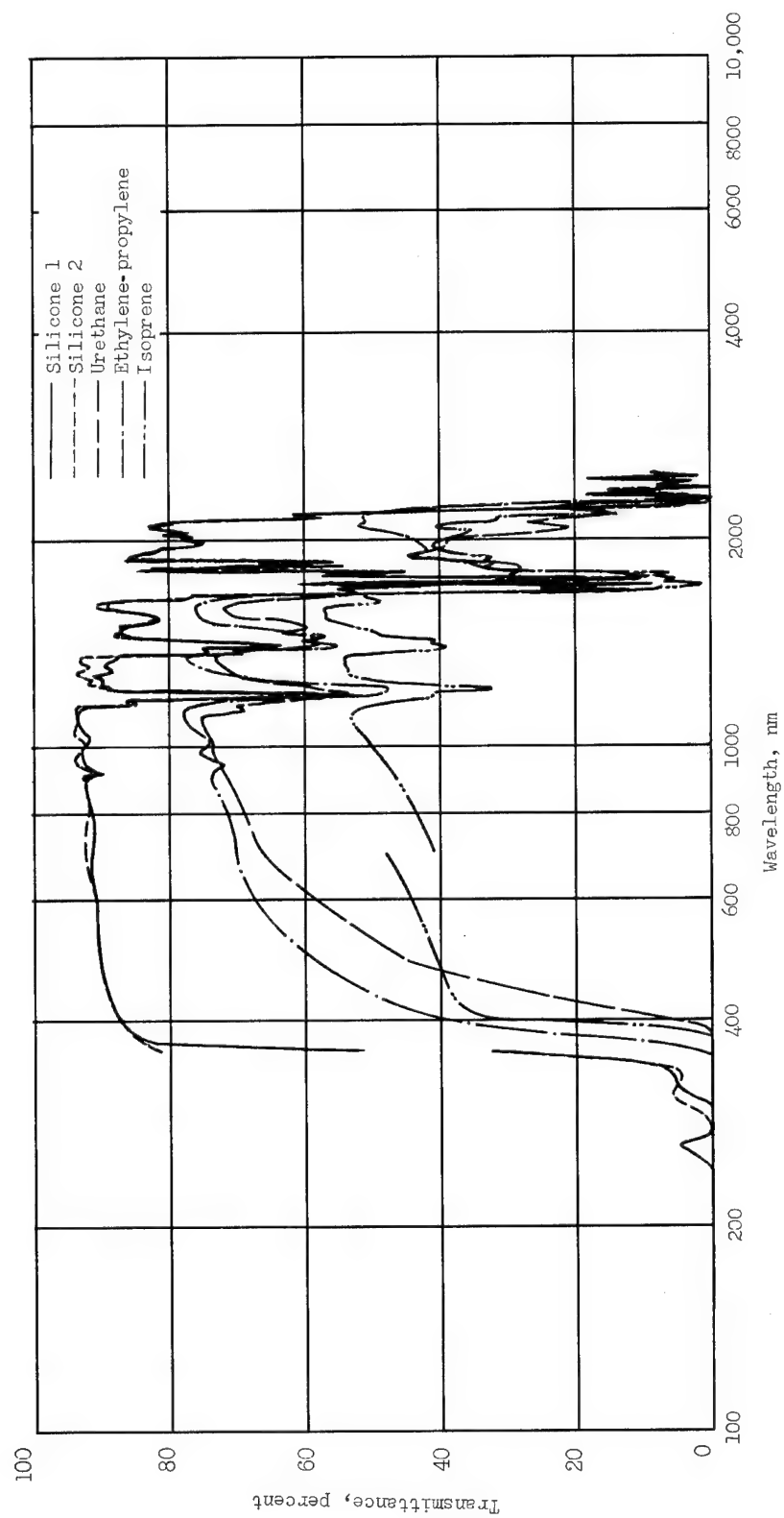


Figure 9.- Comparison of the transmittance of the elastomers.



(a) Chart only.



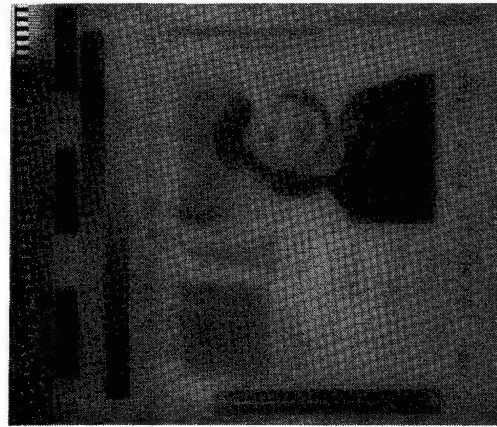
(b) Silicone 1.



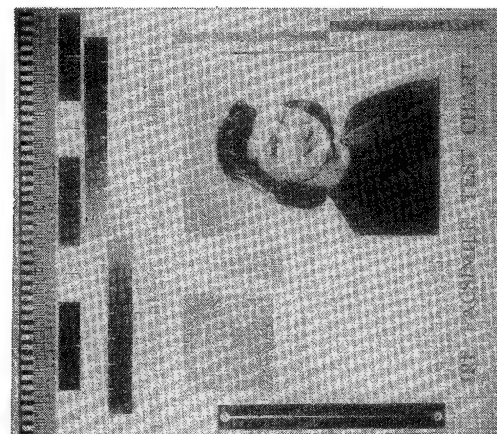
(c) Silicone 2.



(d) Urethane rubber.



(e) Ethylene-propylene rubber.



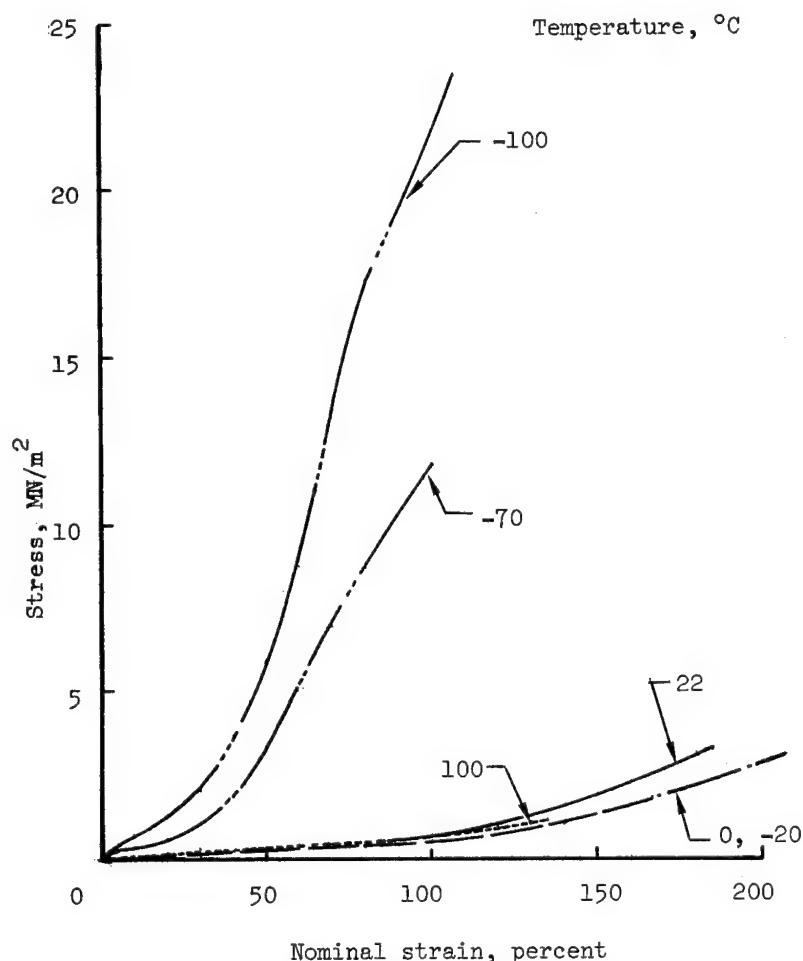
(f) Isoprene rubber.
L-71-501

Figure 10.- Photographs of a test chart with transparent elastomer sheets between the camera and the chart.

wrinkling. The image transmitted by silicones 1 and 2 are very close in clarity and contrast to that without the intervening panel. Considerable loss of contrast as well as darkening (indicative of reduced transmittance) appears in photographs taken through the urethane, ethylene-propylene, and isoprene rubbers. Improved molding techniques could probably be used to improve the optical clarity of the urethane rubber.

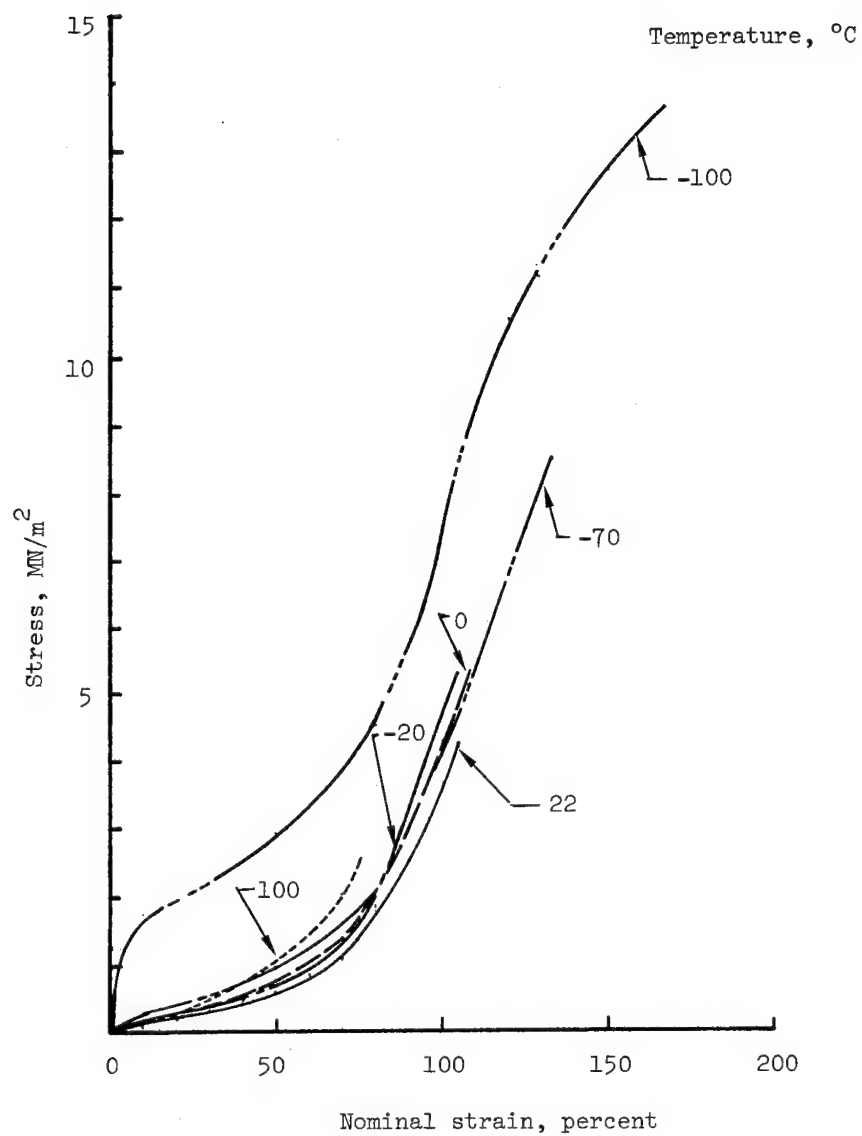
Effect of Temperature on Elastomers

Mechanical properties.- Representative values of the variation of stress with nominal strain at selected temperatures between -100°C and $+100^{\circ}\text{C}$ are shown in figure 11. At -100°C all the elastomers except the silicone rubbers have a comparatively high modulus, and the strain at which failure occurs is reduced two orders of magnitude



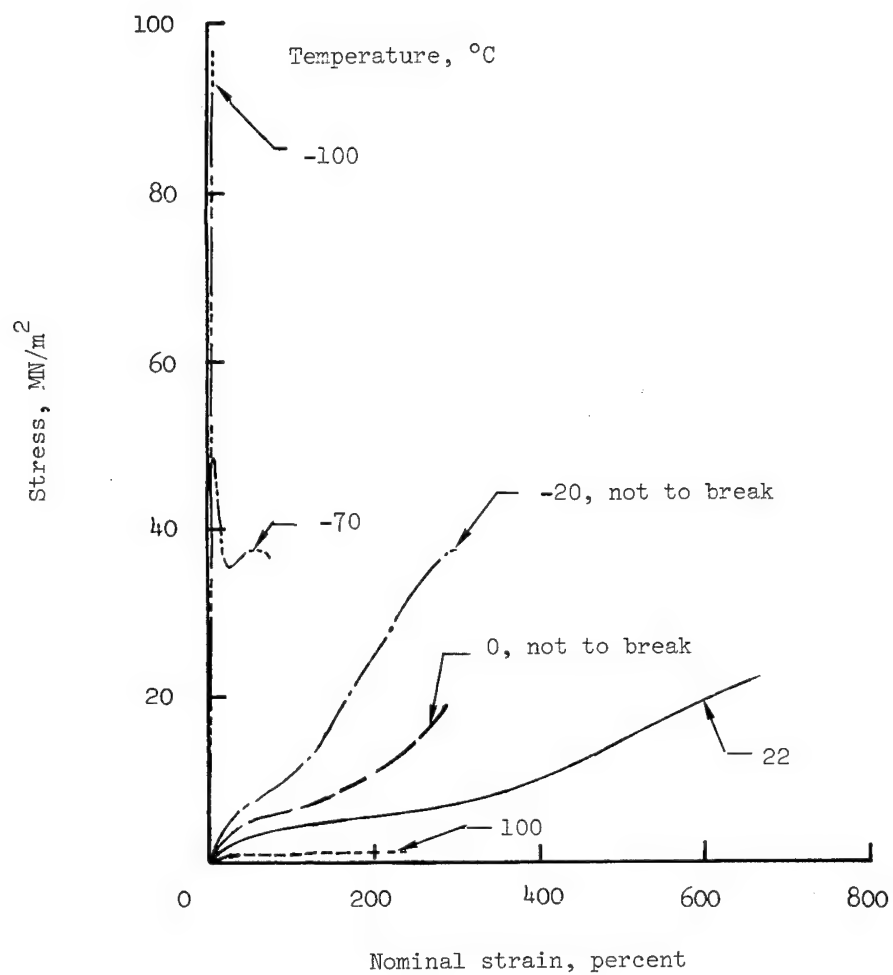
(a) Silicone 1.

Figure 11.- Variation of stress and nominal strain at an elongation rate of 0.254 m/min at selected test temperatures.



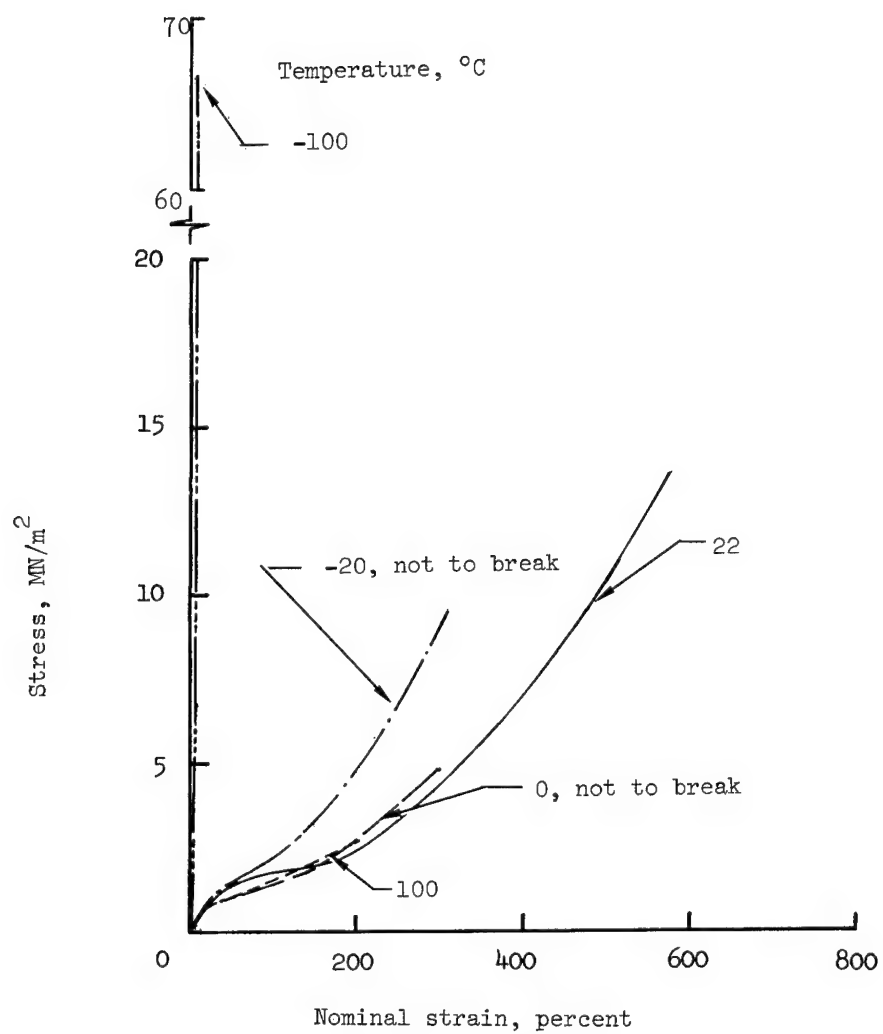
(b) Silicone 2.

Figure 11.- Continued.



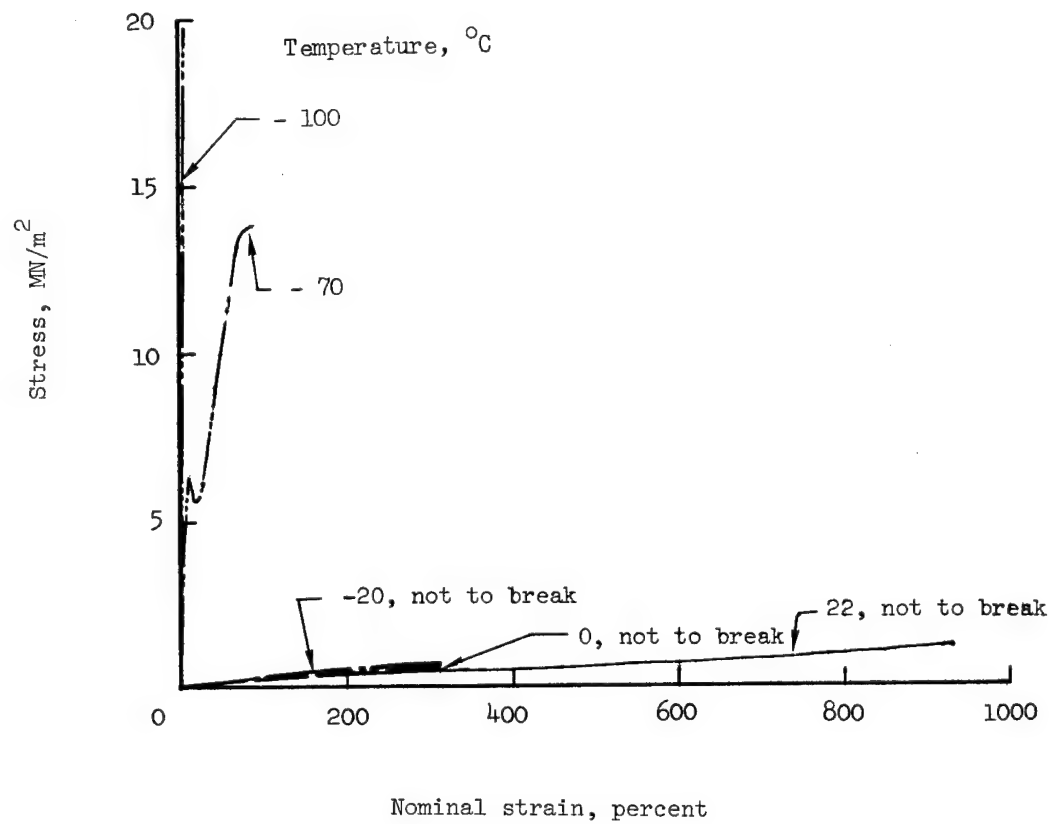
(c) Urethane rubber.

Figure 11.- Continued.



(d) Ethylene-propylene rubber.

Figure 11.- Continued.



(e) Isoprene rubber.

Figure 11.- Concluded.

below that at ambient laboratory conditions. At -70°C , the urethane and isoprene rubbers exhibit an upper and lower yield point similar to that experienced by some metals. The data of figures 11(a) and 11(b) indicate that both silicone rubbers were tested at temperatures above the glass transition temperature. At 100°C , all the elastomers failed at lower strains and stresses than at ambient laboratory conditions. (See table VI.)

TABLE VI.- ELASTOMER PROPERTIES AT BREAK FOR SELECTED TEMPERATURES^a

[Number following \pm is one standard deviation]

| Material | Ultimate stress, MN/m ² | | Ultimate nominal strain, percent | | Energy at break, joules | |
|--------------------|------------------------------------|------------------------------|----------------------------------|-----------------------------|------------------------------|------------------------------|
| | 23 ^o C | 100 ^o C | 23 ^o C | 100 ^o C | 23 ^o C | 100 ^o C |
| Silicone 1 | 3.15 \pm 0.16 | 0.894 \pm 0.264 | 165 \pm 13 | 128 \pm 19 | 1.32 \pm 0.22 | 0.47 \pm 0.17 |
| Silicone 2 | 3.29 \pm 0.54 | ^b 1.83 \pm 0.35 | 98 \pm 10 | ^b 80.0 \pm 5.1 | 0.94 \pm 0.35 | ^b 0.54 \pm 0.14 |
| Urethane | 23.2 \pm 1.8 | 0.885 \pm 0.120 | 734 \pm 39 | 239 \pm 51 | 535.7 \pm 40.1 | 1.91 \pm 0.63 |
| Ethylene-propylene | 12.7 \pm 1.0 | ^b 1.70 \pm 0.18 | 534 \pm 29 | ^b 187 \pm 17 | 260.0 \pm 29.1 | ^b 2.38 \pm 0.47 |
| Isoprene | ^c 1.19 \pm 0.05 | ----- | ----- | ----- | ^c 4.84 \pm 0.30 | ----- |

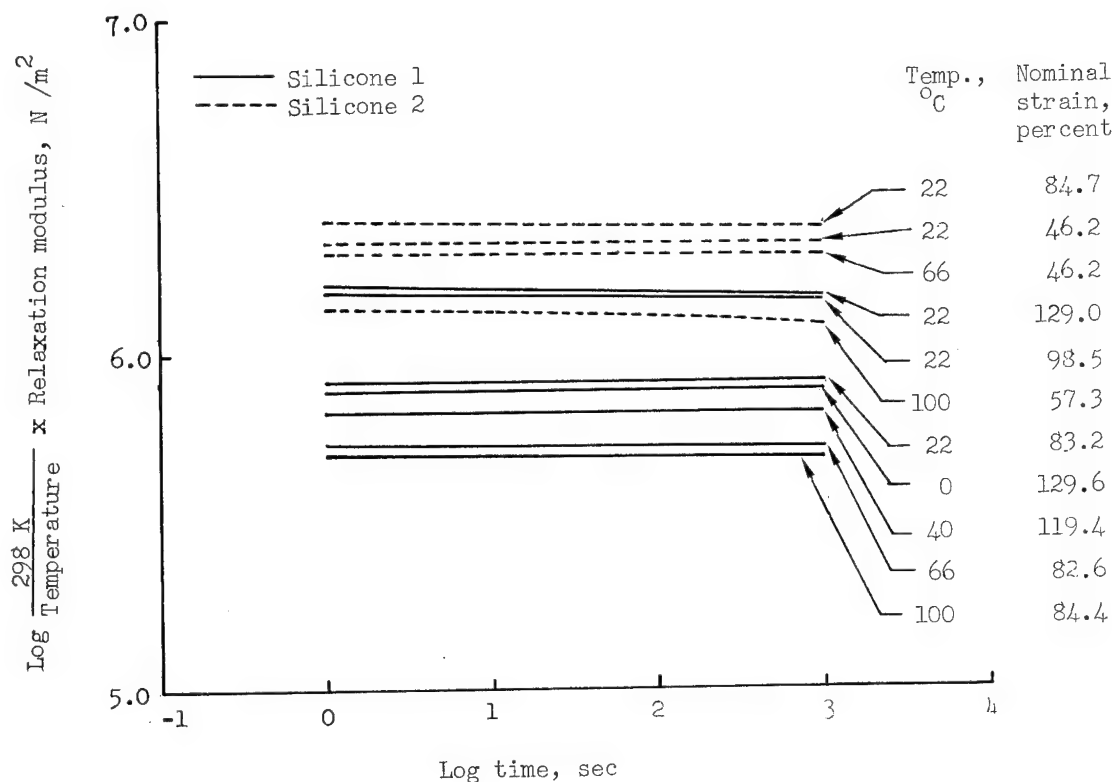
^aAverage of 10 tests except as noted.

^bAverage of 9 tests.

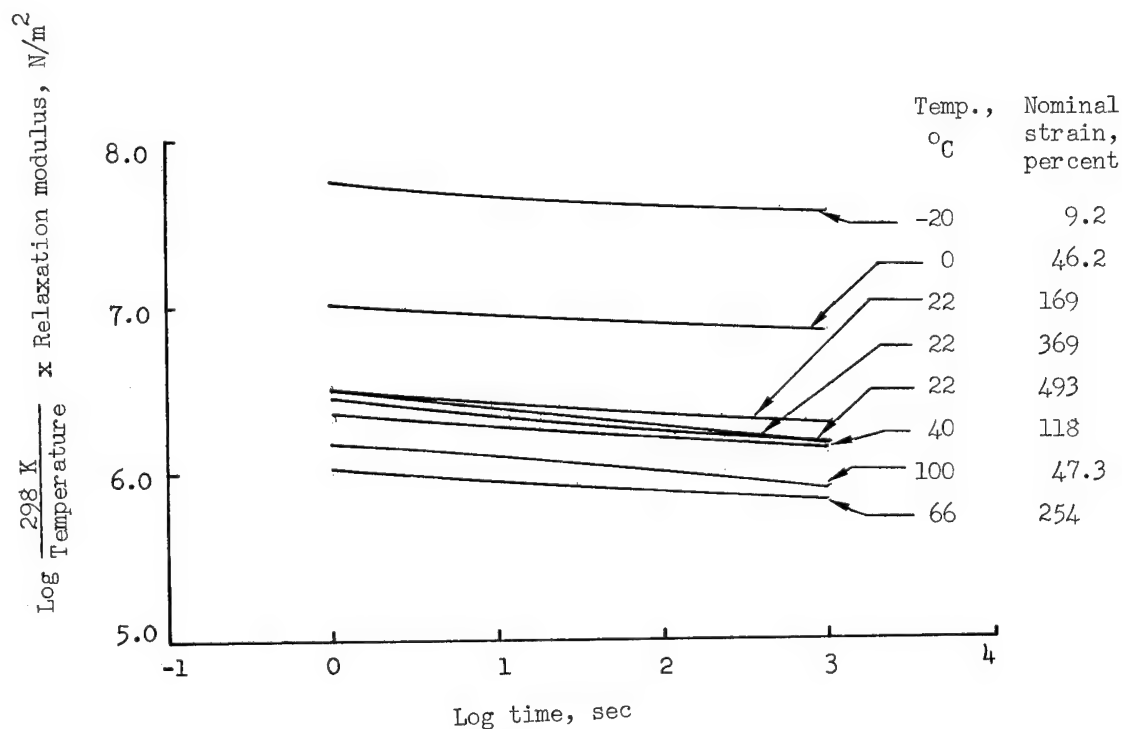
^cProperties at strain of 934 percent without break.

Stress relaxation.- Stress relaxation test results are presented in figure 12 for each of the five elastomers studied. At elevated temperatures, tensile failures of some of the specimens occurred either during initial elongation or during relaxation. In these cases, the magnitude of the load was reduced on successive tests until a full 1000-second duration test was achieved. These materials are not linearly viscoelastic; this fact accounts for the variation in response of the curves at the same temperature, but at different starting loads.

In general, there are five distinct regions of viscoelastic behavior for polymeric materials: glassy, transition, rubbery, rubbery flow, and liquid flow regions. (See refs. 10 to 13.) The mechanical behavior in the temperature ranges indicated in figure 12 of both silicone rubber materials as well as the isoprene is a rubbery-type response as indicated by the negligible change in modulus response with time. The urethane and ethylene-propylene rubbers exhibit behavior characteristic of the latter stages of the transition region between the glassy and rubbery regions. At elevated temperatures, the ethylene-propylene rubber exhibits rubbery and the initial stage of rubbery flow behavior.

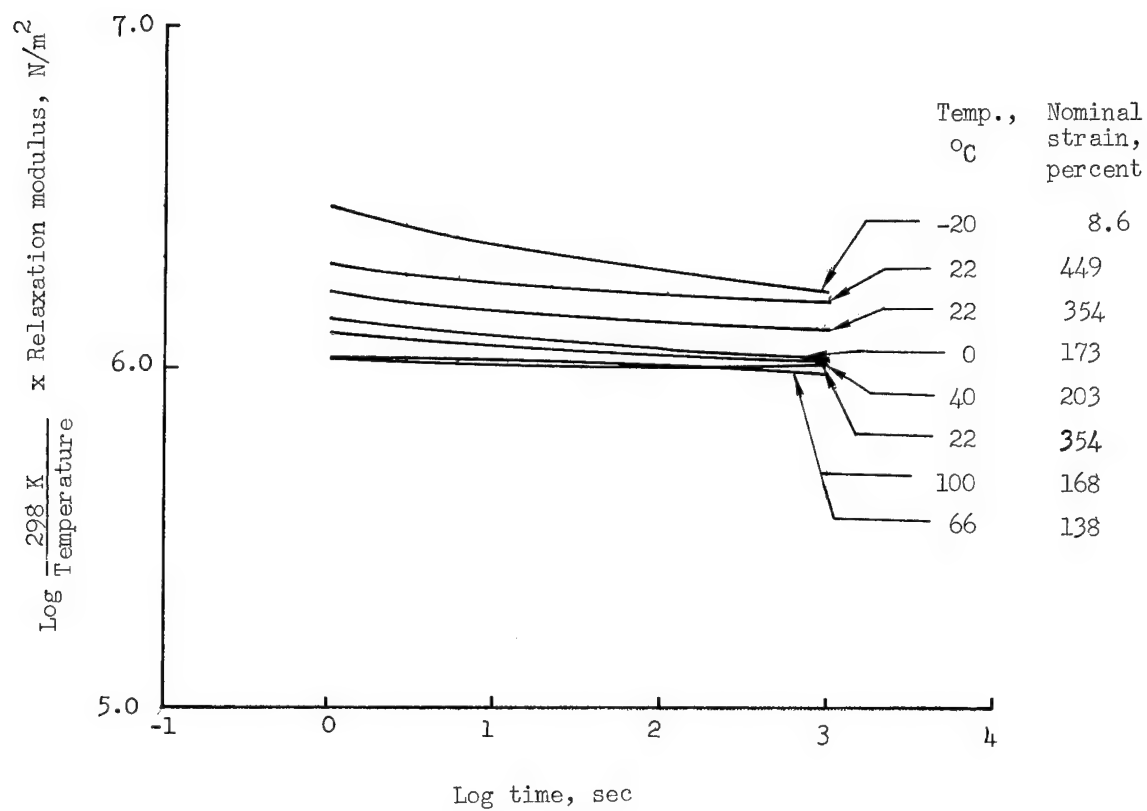


(a) Silicone rubber.

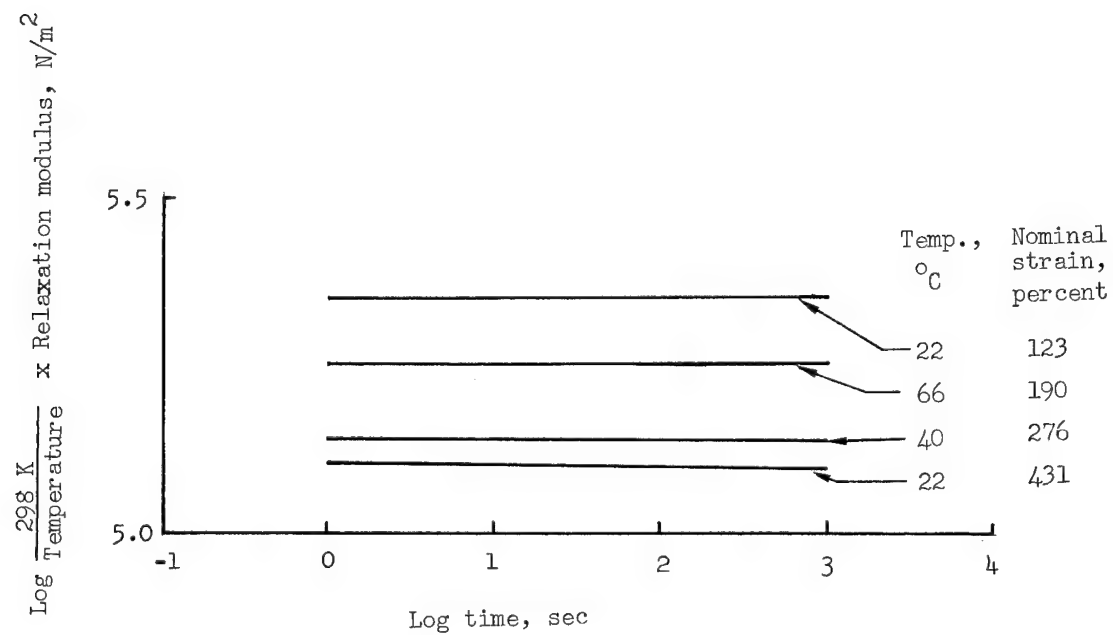


(b) Urethane rubber.

Figure 12.- Stress relaxation test results for the elastomers at selected temperatures.



(c) Ethylene-propylene rubber.



(d) Isoprene rubber.

Figure 12.- Concluded.

Thermal degradation.- Thermograms for each of the elastomers are presented in figure 13. These thermograms show the temperatures at which exothermic reactions occur and provide an indication of the severity and mechanism of the reaction. These data show that in an oxygen atmosphere, both silicone rubber materials have an exothermic reaction at 275°C , the urethane and ethylene-propylene rubbers at 200°C , and the isoprene rubber at 120°C . Comparison of the thermograms in oxygen and nitrogen allow one to predict that none of the materials can be regarded as nonflammable in a 100-percent oxygen environment at 100 kN/m^2 pressure.

Test specimens of urethane rubber and isoprene rubber melted when subjected to a temperature of 120°C in an oven. Ethylene-propylene rubber under the same exposure became dark and opaque without melting. Subsequent microscopic examination of the ethylene-propylene rubber specimen showed that crystallization had occurred.

Effects of Simulated Space Environment Factors

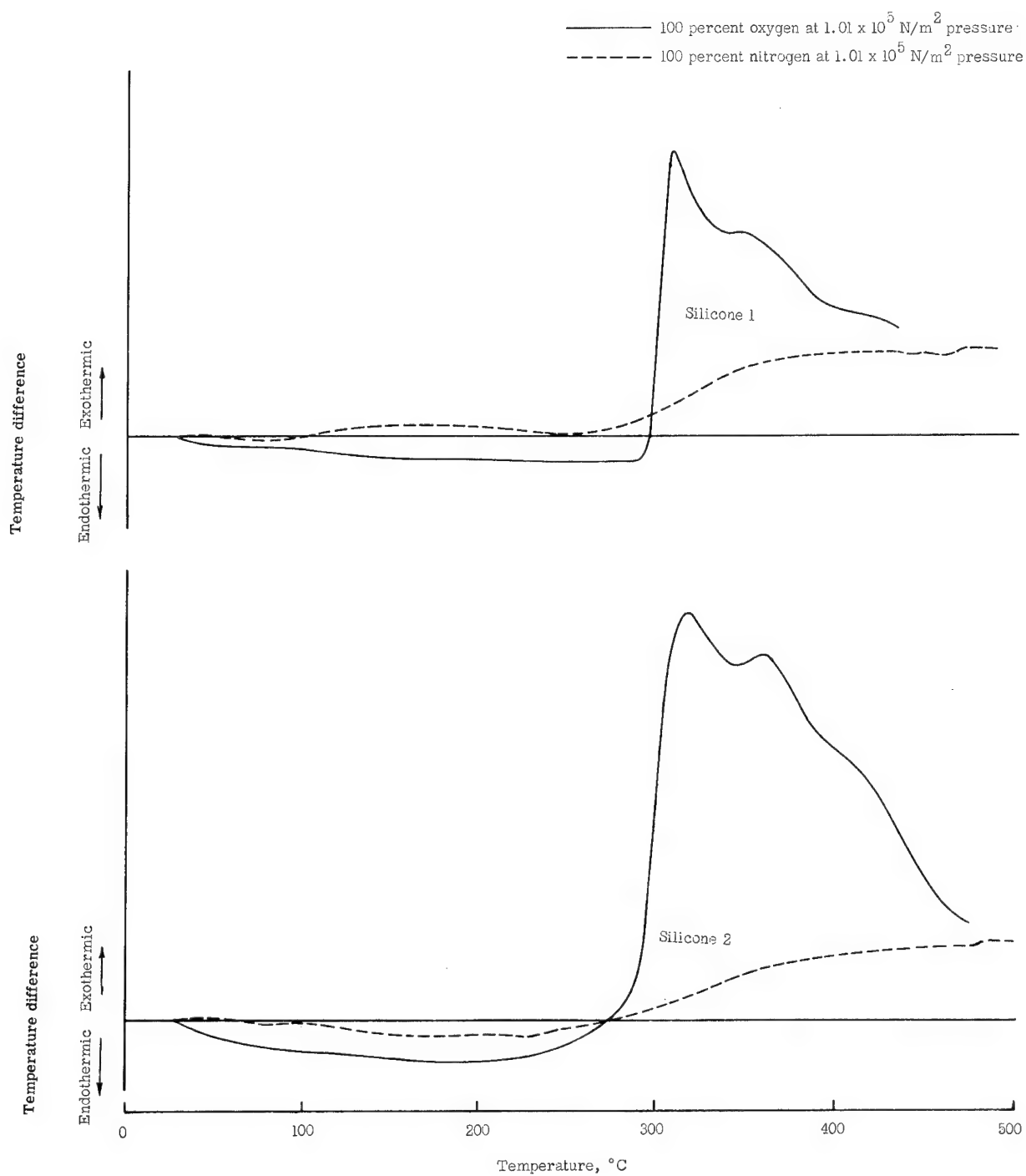
Mechanical properties.- The loading response of the materials studied in this investigation after exposure to selected environmental conditions are presented in figure 14. The graph for stress against nominal strain for a control specimen exposed to air only is also given in each of these graphs for comparison.

The air-heat exposure ($100^{\circ} \pm 3^{\circ}\text{C}$ for 30 days) increased the modulus (ratio of stress to strain) for both of the silicone rubber materials, but had little effect on the modulus of the urethane rubber and lowered the modulus for the ethylene-propylene rubber. The ethylene-propylene rubber, for all practical purposes, was not useful for structural or optical applications after this exposure condition. It became opaque and brittle and lost much of its ability to elongate and carry load. Results for isoprene rubber are not shown because the material melted under air-heat exposure.

After vacuum exposure ($1.3\text{ }\mu\text{N/m}^2$ for 31 days), the modulus of all the materials except silicone 1 and isoprene rubber increased. Very little change in modulus occurred for silicone 1 and isoprene rubber.

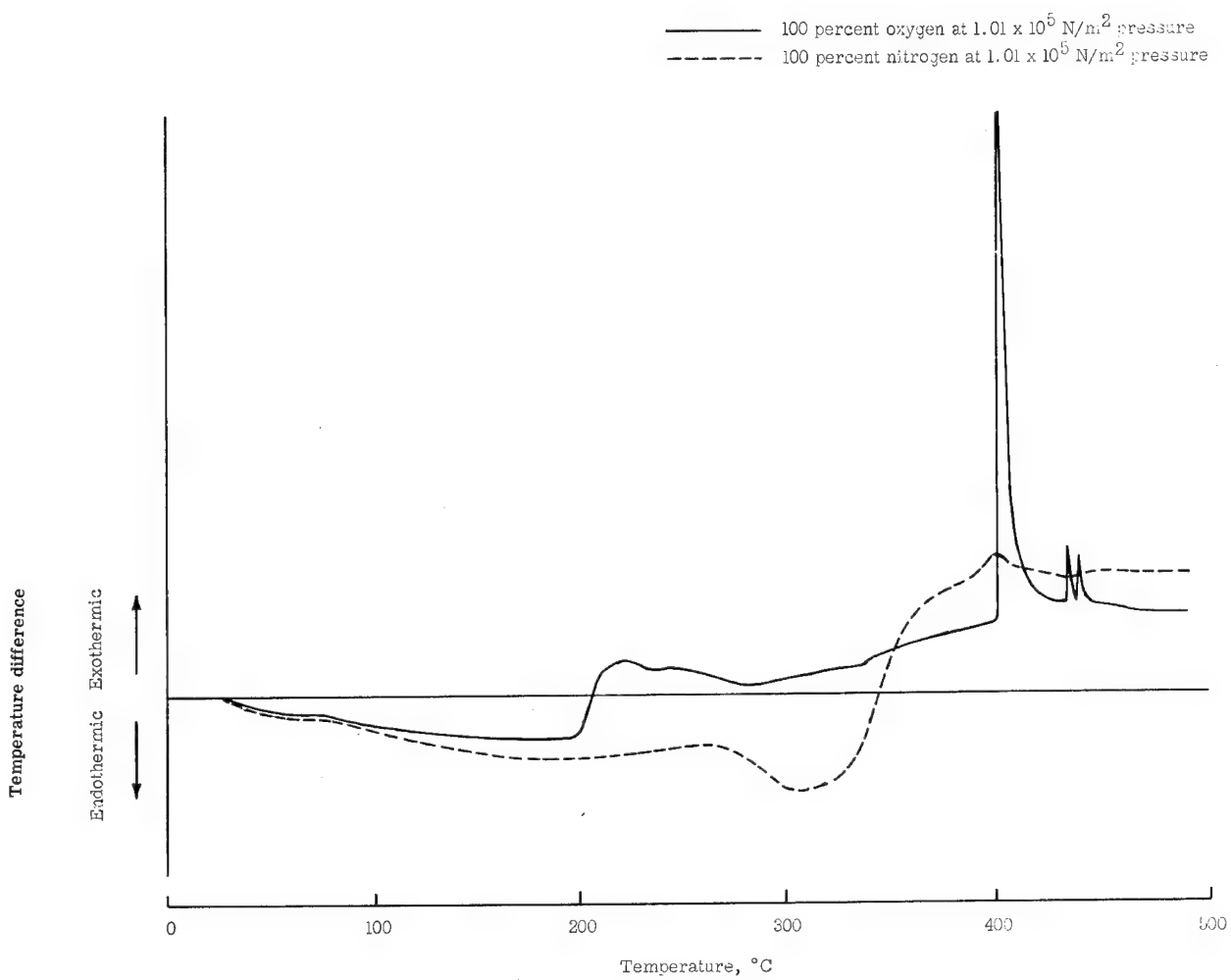
Exposure of the materials to vacuum-heat conditions ($100^{\circ} \pm 4^{\circ}\text{C}$ and $130\text{ }\mu\text{N/m}^2$ for 30 days) resulted in a decrease in modulus for silicone 1 and an increase in modulus for silicone 2 and urethane rubber. Vacuum-heat tests were not conducted on the isoprene and ethylene-propylene materials because of their low melting temperatures.

Data for the ultimate stress, ultimate nominal strain, and work required to break the specimen after exposure to air, air heat ($100^{\circ} \pm 3^{\circ}\text{C}$ for 30 days), vacuum ($1.3\text{ }\mu\text{N/m}^2$ for 31 days), and vacuum heat ($130\text{ }\mu\text{N/m}^2$ and $100^{\circ} \pm 4^{\circ}\text{C}$ for 30 days) are presented in table VII.



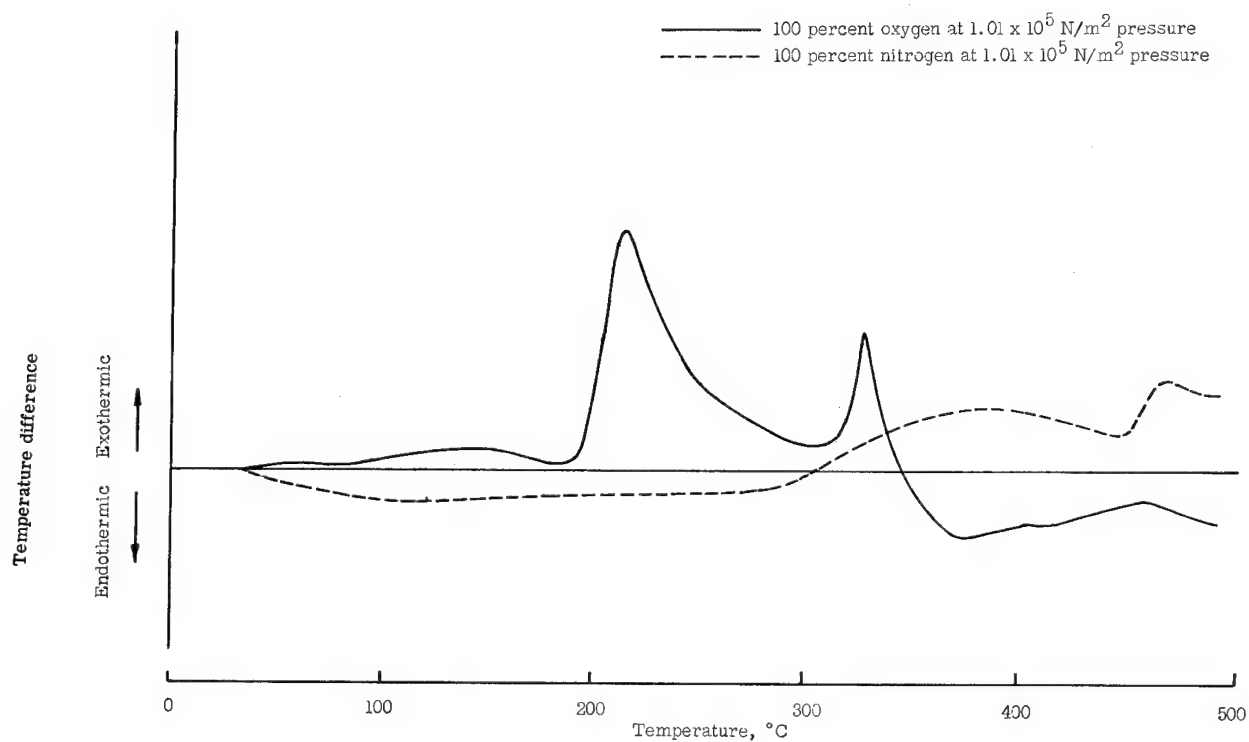
(a) Silicone rubbers.

Figure 13.- Differential thermal analysis thermograms of elastomers.



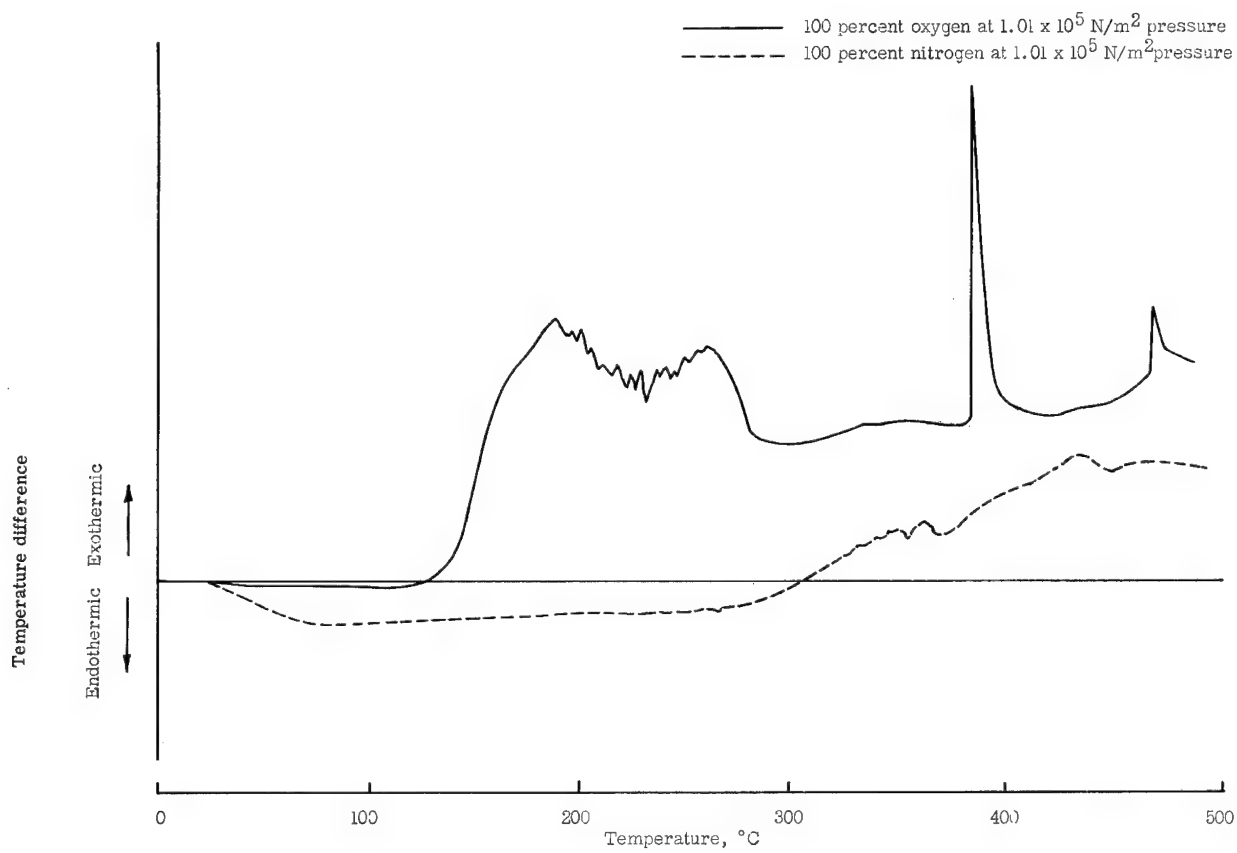
(b) Urethane rubber.

Figure 13.- Continued.



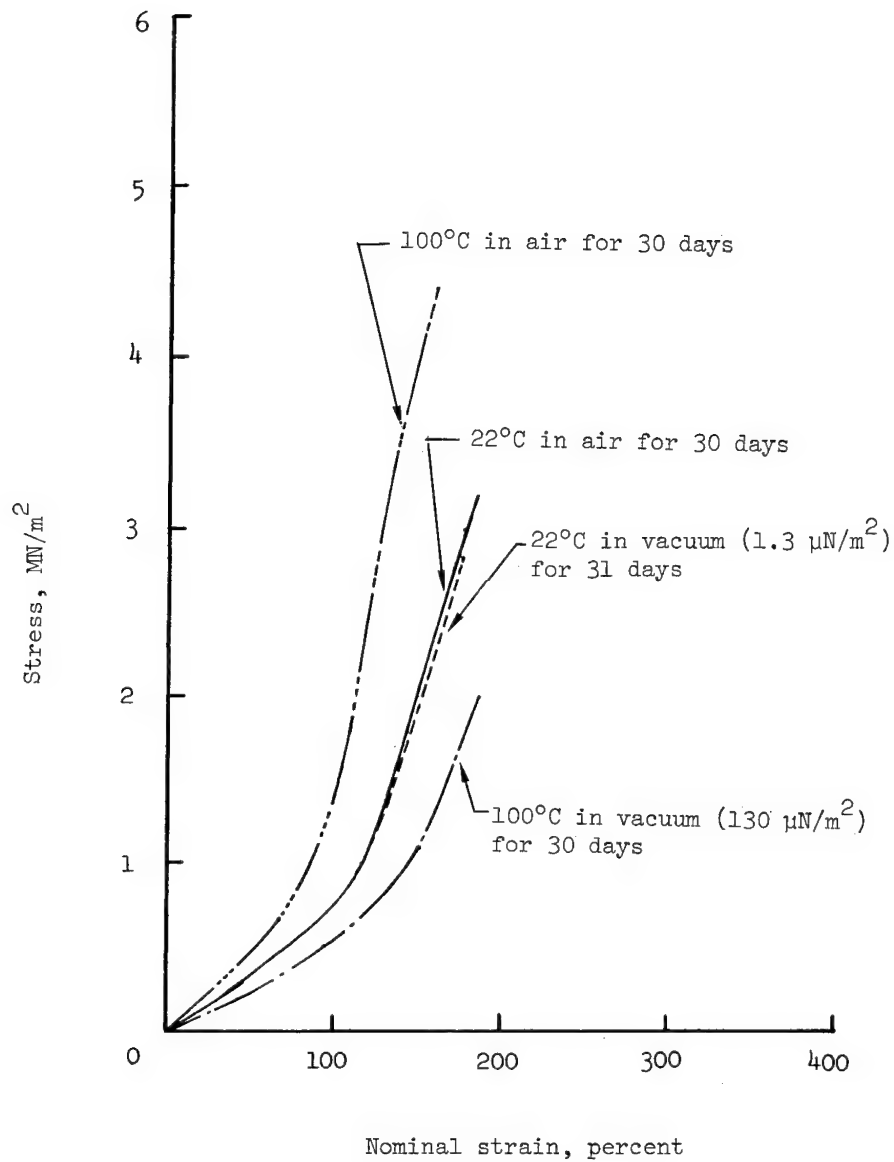
(c) Ethylene-propylene rubber.

Figure 13.- Continued.



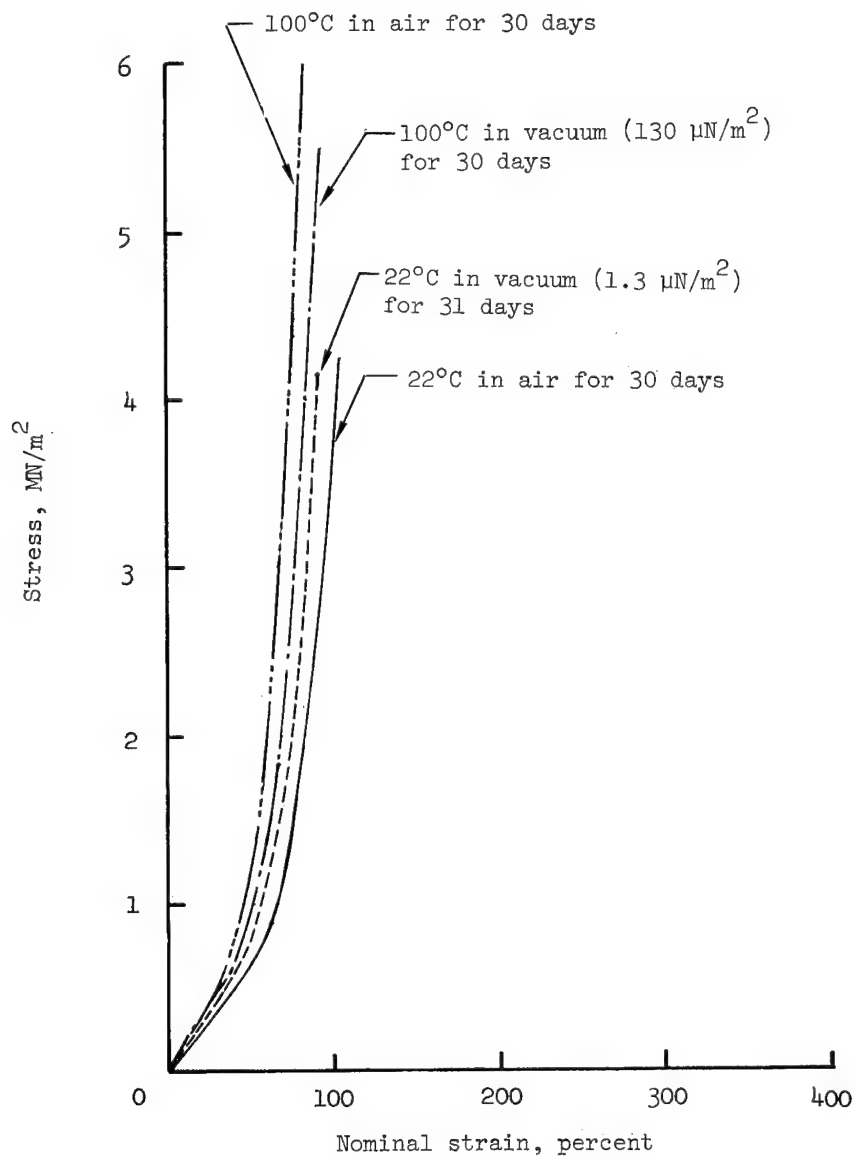
(d) Isoprene rubber.

Figure 13.- Concluded.



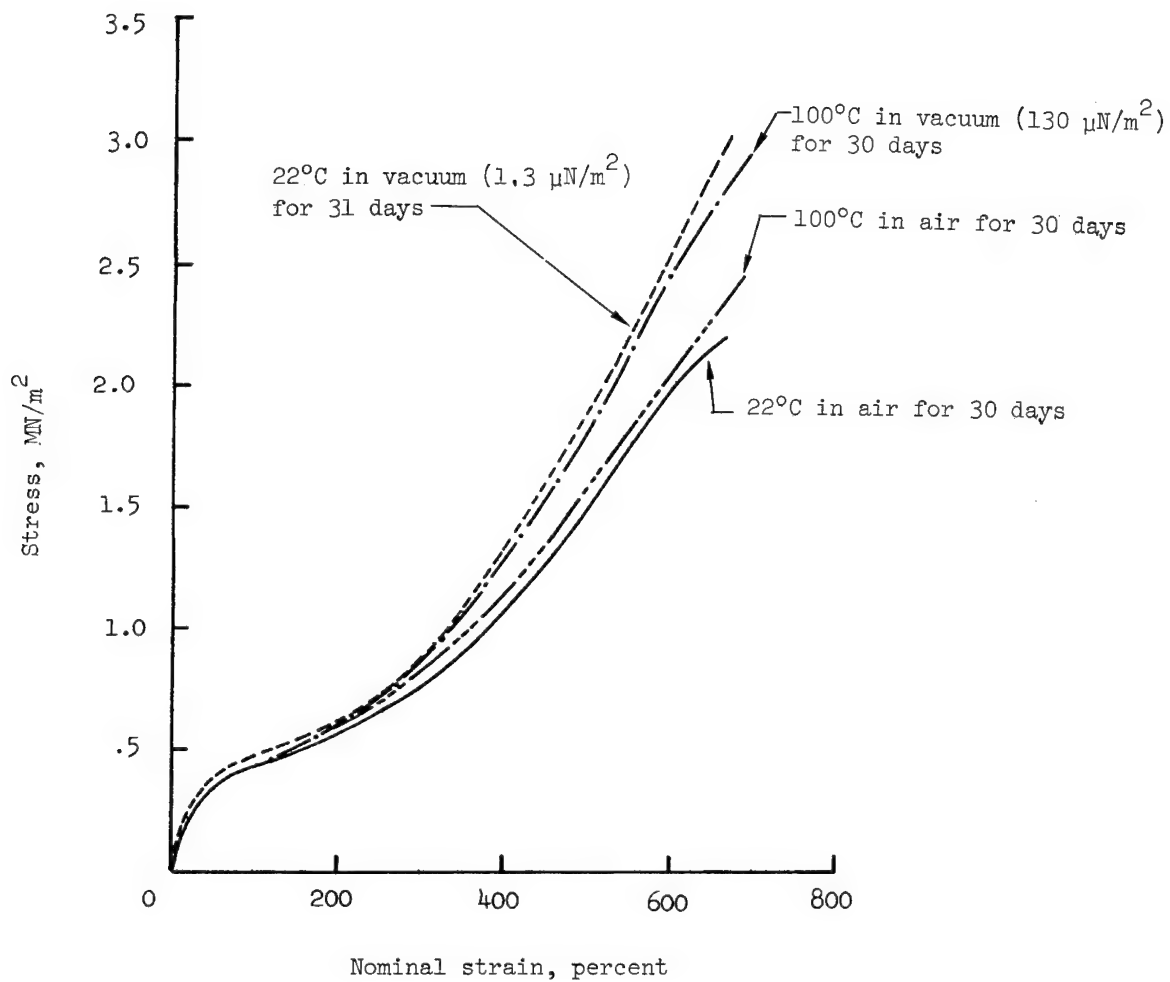
(a) Silicone 1.

Figure 14.- Effect of environmental exposure on the stress and nominal strain of the elastomers. Specimen loaded at ambient laboratory conditions.



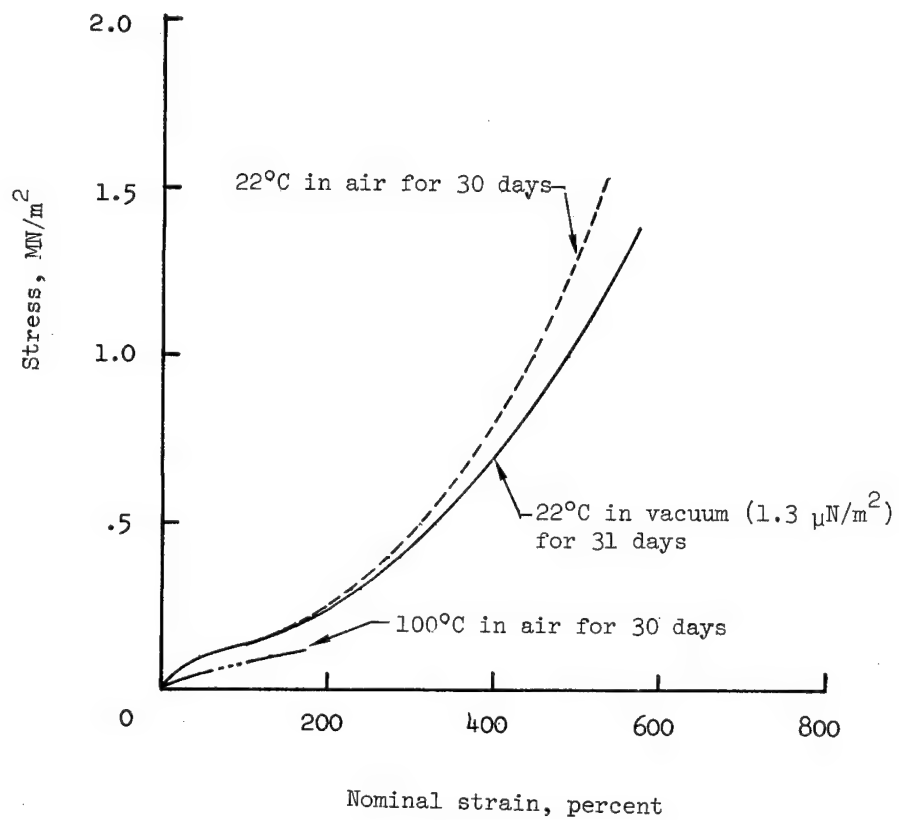
(b) Silicone 2.

Figure 14.- Continued.



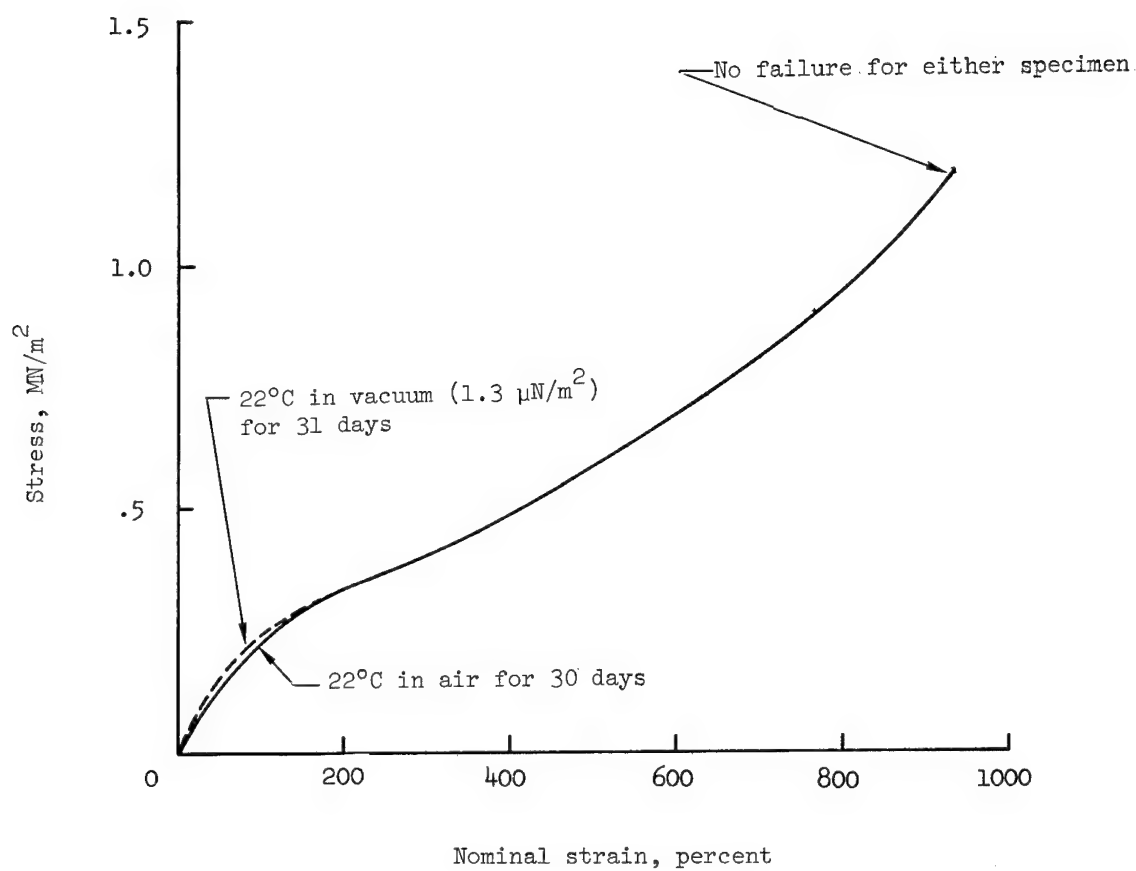
(c) Urethane rubber.

Figure 14.- Continued.



(d) Ethylene-propylene rubber.

Figure 14.- Continued.



(e) Isoprene rubber.

Figure 14.- Concluded.

TABLE VII.- ELASTOMER PROPERTIES AT BREAK FOLLOWING EXPOSURE TO SELECTED ENVIRONMENTS^a[Number following \pm is one standard deviation]

| Elastomer | Ultimate stress, MN/m ² | | | | Ultimate nominal strain, percent | | | | Energy at break, joules | | | |
|-----------------------|------------------------------------|------------------------------|-----------------|------------------------|----------------------------------|--------------------------|--------------|-----------------|-------------------------|--------------------------------|-------------------|-------------------|
| | Air (b) | Air-heat (c) | Vacuum (d) | Vacuum- heat (e) | Air | Air-heat | Vacuum | Vacuum- heat | Air | Air-heat | Vacuum | Vacuum- heat |
| Silicone 1 | 3.15 \pm 0.16 | ^f 4.47 \pm 0.32 | 2.47 \pm 0.52 | 1.68 \pm 0.51 | 165 \pm 13 | ^f 136 \pm 5 | 153 \pm 14 | 149 \pm 26 | 1.323 \pm 0.221 | ^f 1.699 \pm 0.246 | 0.973 \pm 0.238 | 0.725 \pm 0.283 |
| Silicone 2 | 3.29 \pm 0.54 | 5.90 \pm 0.62 | 3.35 \pm 0.56 | 5.13 \pm 0.97 | 98 \pm 10 | 77 \pm 3 | 89.2 \pm 5 | 86 \pm 6 | 0.943 \pm 0.348 | 1.089 \pm 0.143 | 0.666 \pm 0.111 | 0.878 \pm 0.244 |
| Urethane | 23.2 \pm 1.8 | ^g 24.0 \pm 1.5 | 31.7 \pm 1.9 | 29.5 \pm 2.5 | 734 \pm 39 | 690 \pm 23 | 685 \pm 17 | 693 \pm 25 | 535.7 \pm 40.1 | ^g 655.7 \pm 48.8 | 736.6 \pm 56.7 | 682.9 \pm 78.1 |
| Ethylene-propylene | 12.7 \pm 1.0 | ^h 1.20 \pm 0.14 | 14.9 \pm 1.6 | 15.6 \pm 1.3 | 534 \pm 29 | 164 \pm 22 | 538 \pm 29 | 488 \pm 18 | 260.0 \pm 29.1 | ^h 9.0 \pm 2.3 | 225.6 \pm 35.2 | ----- |
| Isoprene ⁱ | 1.19 \pm 0.05 | ----- | 1.14 \pm 0.05 | ----- | ----- | ----- | ----- | ----- | 4.84 \pm 0.30 | ----- | 4.74 \pm 0.21 | ----- |

^aAverage of 10 tests except as noted.^b22°C for 30 days.^c100°C for 30 days.^d22°C, 1.3 μ N/m² for 31 days.^e100°C, 130 μ N/m² for 30 days.^fAverage of 7 tests.^gAverage of 9 tests.^hAverage of 8 tests.ⁱProperties at strain of 934 percent without break.

The ultimate strength of silicone 1 was increased by exposure to air heat and decreased as a result of exposure to vacuum and vacuum-heat conditions (relative to the air control group). Air-heat, vacuum, and vacuum-heat exposure increased the ultimate strength of the silicone 2 and urethane rubber materials. The ultimate strength of the ethylene-propylene rubber increased after vacuum and vacuum-heat exposure, but decreased after air-heat exposure. The presence of air during heating caused a chemical reaction and changed the initial properties of this material. The data indicate that the selected environment conditions have no significant influence on the ultimate nominal strain for the test materials with the exception of air-heat exposure on the ethylene-propylene rubber; in that case it was reduced.

The energy required to break the urethane rubber was more than two orders of magnitude greater than that for the silicone or isoprene rubbers and over twice as great as that for the ethylene-propylene rubber.

Elastomer weight changes.— Graphical representations of the weight loss with time of exposure at room temperature in a vacuum (130 μ N/m²) for the five elastomers are presented in figure 15. The percent changes in weight as a result of various environmental exposures (air-heat, vacuum, and vacuum-heat) for these materials are presented in table VIII. The data from table VIII for vacuum exposure are also represented by symbols in figure 15.

It is common to assume in cursory screening that polymeric materials which lose less than 1 percent of their weight in a thermal vacuum environment are likely to be suitable for spacecraft applications. (See ref. 14.) None of the elastomers tested satisfy this criterion after vacuum-heat exposure. The silicone rubbers, however, did lose less than 1 percent of their initial weight after air-heat and vacuum exposure. The

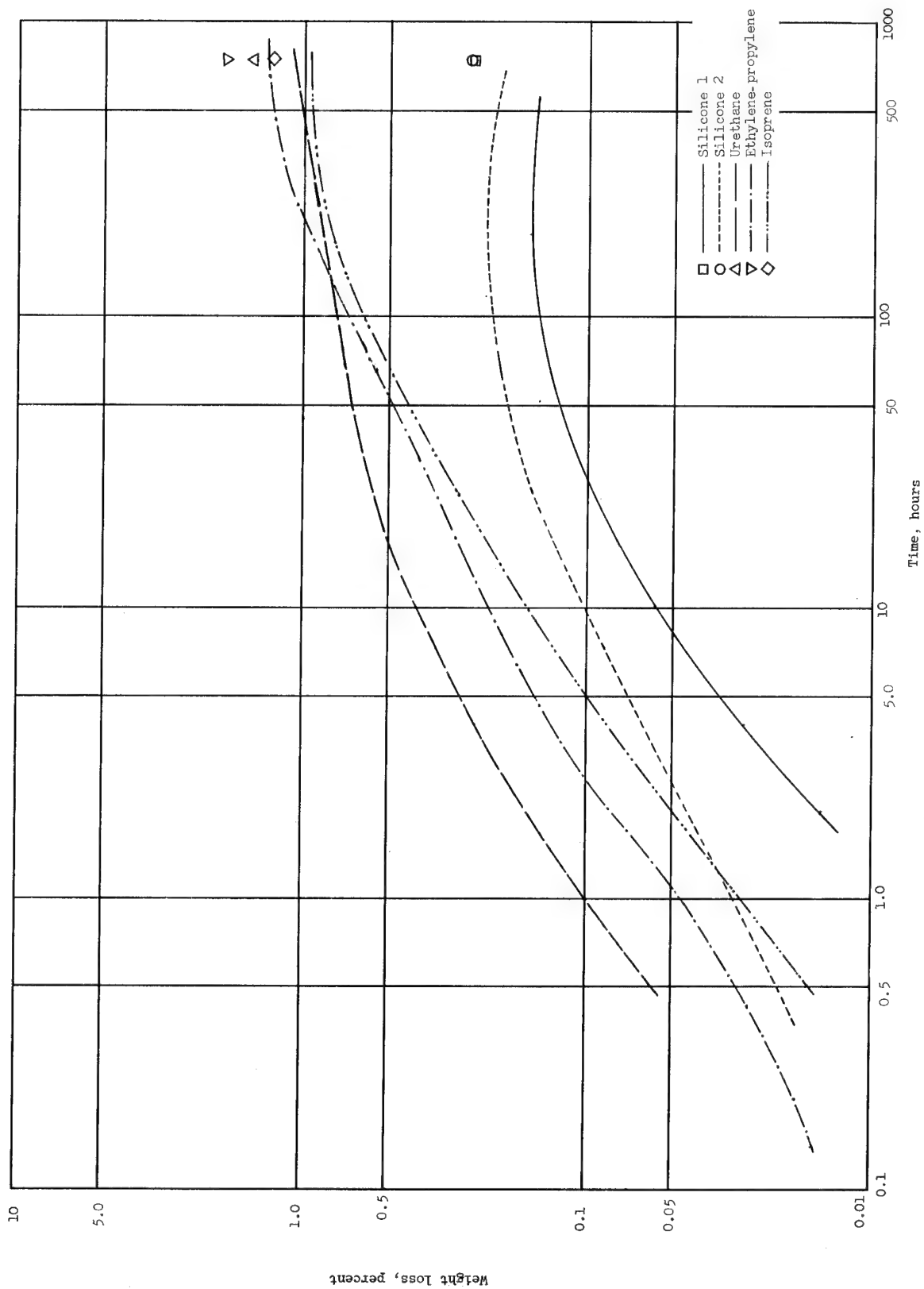


Figure 15.- Vacuum weight loss of elastomers. Lines represent "in situ" data for a pressure of 130 $\mu\text{N}/\text{m}^2$. Symbols denote results for test specimens exposed to vacuum (1.3 $\mu\text{N}/\text{m}^2$), but weighed in air.

TABLE VIII.- WEIGHT LOSS DATA FOR ELASTOMERS AFTER
EXPOSURE TO SELECTED ENVIRONMENTS^a

[Number following \pm is one standard deviation]

| Elastomer | Weight loss, percent | | |
|--------------------|----------------------|--------------------------------|--------------------------------|
| | Air-heat (b) | Vacuum (c) | Vacuum-heat (d) |
| Silicone 1 | 0.450 \pm 0.078 | ^e 0.253 \pm 0.011 | 2.548 \pm 1.550 |
| Silicone 2 | 0.375 \pm 0.035 | 0.258 \pm 0.024 | 1.508 \pm 0.044 |
| Urethane | 1.475 \pm 0.047 | 1.529 \pm 0.040 | ^e 2.473 \pm 0.021 |
| Ethylene-propylene | -0.068 \pm 0.115 | 1.935 \pm 0.012 | 3.559 \pm 0.128 |
| Isoprene | ----- | 1.355 \pm 0.109 | ----- |

^aAverage of 10 tests except as noted.

^b100° C for 30 days.

^c22° C, 1.3 μ N/m² for 31 days.

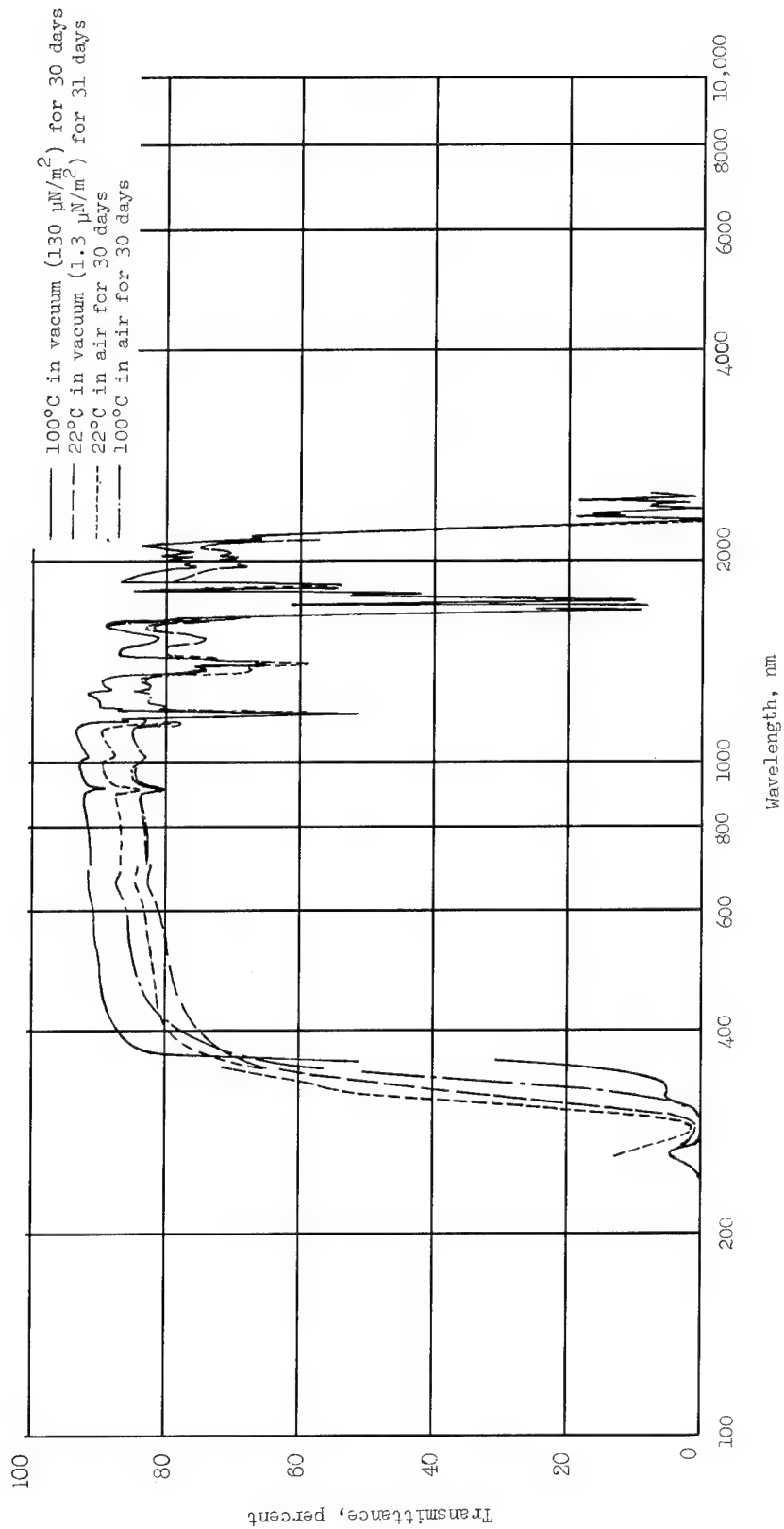
^d100° C, 130 μ N/m² for 30 days.

^eAverage of 9 tests.

insignificant weight change for the ethylene-propylene after air-heat exposure is probably due to a combination of off-gassing (weight loss) and oxidation (weight gain).

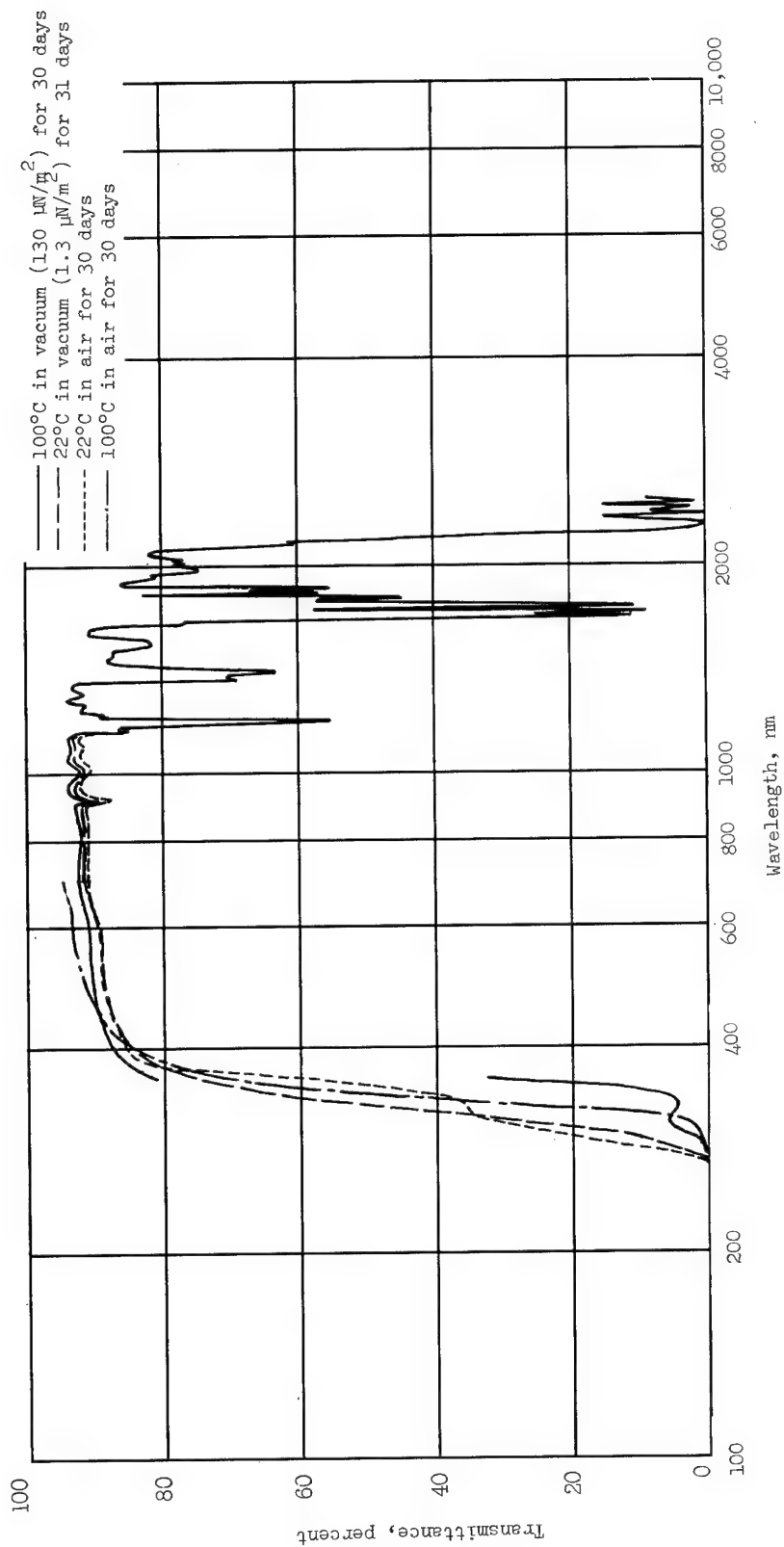
Ultraviolet-vacuum effects.- After the ultraviolet-vacuum exposure of each elastomer, a microscopic examination was made of the surface that was directly exposed to the ultraviolet radiation. There was no evidence of surface degradation as exemplified by surface cracking or crazing in any of the specimens. Photon penetration at a 121.6-nm wavelength would be small compared with specimen thickness and degradation of the material properties due to polymeric chain-scission and cross linking (as measured by uniaxial tension tests of the exposed specimens) was not detected. In similar tests, surface damage to some of these elastomers was reported (ref. 1) after exposure to radiation from 220 nm through the visible range. These tests were conducted in air, however, and the damage was probably caused by photo-oxidation of the surface. Since the anticipated use of these polymers is for windows, permeation of oxygen through the material may produce surface damage when exposed to space radiation. To evaluate this effect further, candidate spacecraft window materials should be test qualified with ultraviolet vacuum on one side and the cabin atmosphere on the other.

Transmittance.- Measurements of the transmittance in the ultraviolet, visible, and infrared spectrum are shown in figure 16 for the five transparent materials after exposure



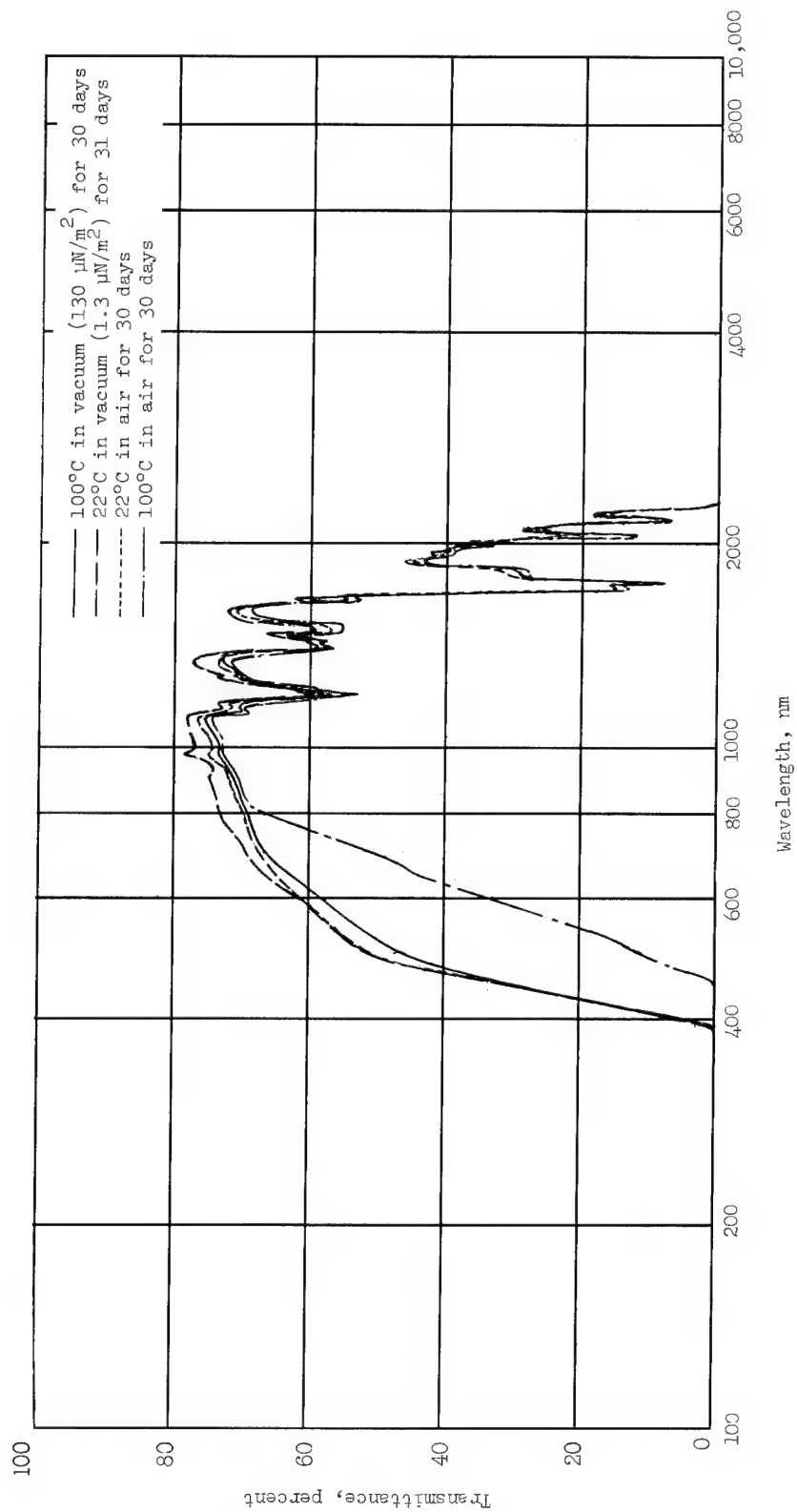
(a) Silicone 1.

Figure 16.- The effect of selected environmental factors on the transmittance of elastomers.



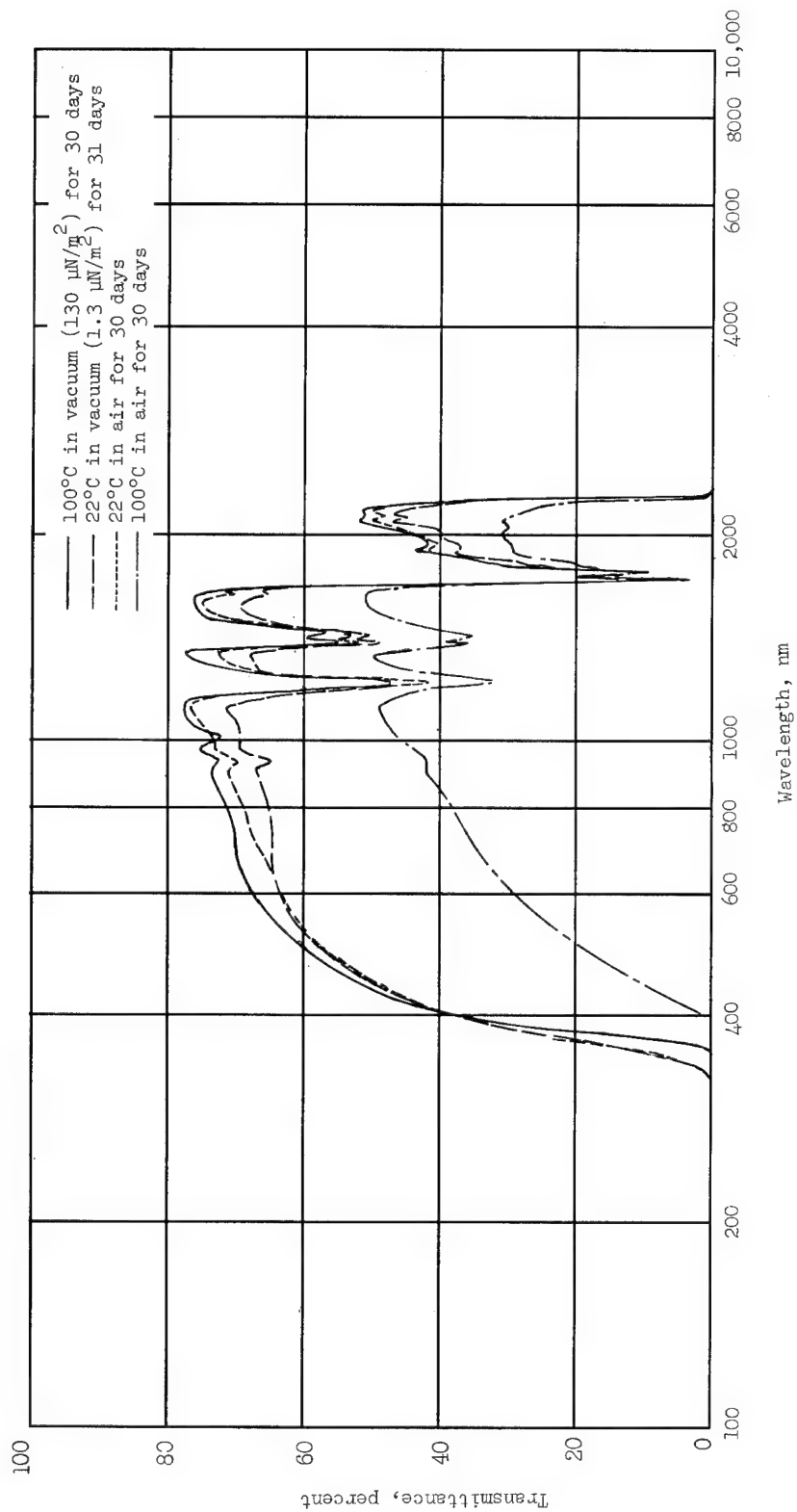
(b) Silicone 2.

Figure 16.- Continued.



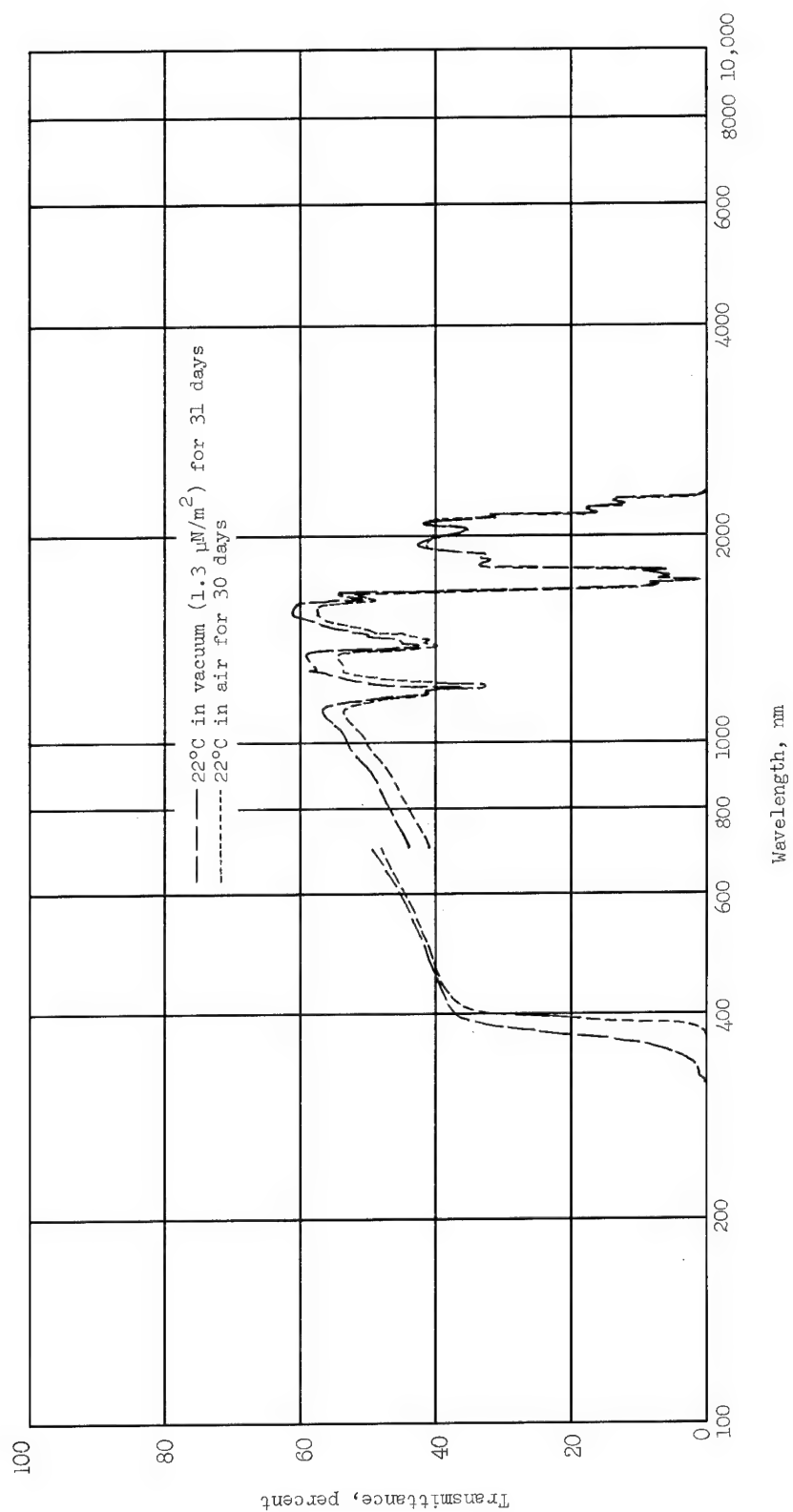
(c) Urethane rubber.

Figure 16.- Continued.



(d) Ethylene-propylene rubber.

Figure 16.- Continued.



(e) Isoprene rubber.

Figure 16.- Concluded.

to an air environment at ambient temperatures, to vacuum ($1.3 \mu\text{N}/\text{m}^2$) at ambient temperatures for 31 days, to vacuum ($130 \mu\text{N}/\text{m}^2$) at a temperature of 100°C for 30 days, and to air at a temperature of 100°C for 30 days. Since isoprene rubber specimens melted at 100°C in air, elevated temperature data for this material were not obtained. The silicone rubbers had a higher transmittance over a broader region of the spectrum than the other elastomers. The experimental data show that exposure to the test environments produced only slight changes in the transmittance of the silicone rubber specimens, whereas exposure to the air-heat environment reduced the transmittance of the urethane appreciably in the visible range and of the ethylene-propylene rubber appreciably over the entire wavelength range.

CONCLUDING REMARKS

An experimental investigation of transparent elastomers which are candidate matrix materials for a flexible window for spacecraft has been made to determine representative values of mechanical, physical, and optical properties over a range of environmental conditions. In addition, some factors which influence the decomposition of the materials were studied. A comparison of data with those measured at ambient conditions of temperature and pressure enables the following comments to be made.

None of the elastomers of this study individually meet the requirements of a spacecraft window matrix under all conditions of space exposure. The ethylene-propylene and isoprene rubbers are inadequate for such application. The silicone and urethane rubbers, however, possess complimentary properties which might profitably be combined in a laminate to satisfy the overall requirements. The urethane rubber possesses good mechanical properties and the silicone rubbers have satisfactory optical properties and resistance to environmental extremes. These properties suggest a composite consisting of an inner core of urethane rubber sheathed on both sides by a thin layer of silicone rubber. In this design, the urethane rubber would act as the main matrix and thus encapsulate the reinforcement filaments. The silicone rubber would provide environmental resistance and improve optical clarity.

Langley Research Center,
National Aeronautics and Space Administration,
Hampton, Va., January 8, 1971.

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